ASSESSMENT OF SPONTANEOUS HEATING OF FRESH AND OXIDIZED COALS

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

BACHELOR OF TECHNOLOGY

IN

MINING ENGINEERING

BY

SANJIB PRAHARAJ

109MN0123

DEPARTMENT OF MINING ENGINEERING

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ROURKELA – 769 008

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UNDER THE GUIDANCE OF

Dr. H. B. SAHU

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National Institute of Technology

Rourkela

CERTIFICATE

This is to certify that the thesis entitled, **—** *"**Assessment of spontaneous heating of fresh and oxidized coals**"* submitted by SriSanjibPraharaj (Roll No. 109MN0123) in partial fulfillment of the requirement for the award of Bachelor of Technology Degree in Mining Engineering at the National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any University/Institute for the award of any Degree or Diploma.

Date:

Dr. H. B. Sahu
Department Of Mining Engineering
National Institute of Technology
Rourkela – 769008

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Date:

SanjibPraharaj
Department of Mining Engineering
National Institute of Technology
Rourkela-769008
2013

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ABSTRACT

Self-heating of coal leading to spontaneous combustion is the most significant cause of fires throughout the world. These fires not only destroy huge coal reserves but also endanger the life of the persons working, towns living above or near the mine and lastly but not least environmental damages occurring due to these fires are incomparable.

Coal oxidation is a natural phenomenon,It may begin in the pit itself or during the operations of extraction, preparation, transportation and storage. A fresh coal is more reactive to oxygen than a weathered coal i.e. it is more liable to spontaneous heating. To see the rate of decrease in the oxidation rate, first coal samples weretaken and three samples were prepared from each sample. First sample was same as the sample taken while the second sample was obtained by oxidizing the sample at 200^0C for 1 day. Third sample was obtained by oxidizing the coal at 200^0C for 3 day. Then susceptibility of each of these samples were checked by crossing point temperature method, differential thermal analysis and wet oxidation potential method. For the experimentation purpose total of 15 samples were taken. Among these 6 samples were from SECL, 2 samples were from MCL, 2 samples were from CCL, 3 samples were from SCCL. 2 samples were collected from Indonesia to compare it's characteristics with the Indian coals.

The intrinsic property of both the fresh and oxidized coal was determined by proximate analysis. Similarly susceptibility of both fresh and oxidized coal to spontaneous heating was calculated by crossing point temperature and wet oxidation potential. The results are tabulated below.

Table 1 Result of proximate analysis, CPT and EMF values of fresh coal samples

SlNo.	Sample	Intrinsic Properties					CPT (^{0}C)	EMF (mV)
		M (%)	VM (%)	A (%)	FC (%)	Calorific Value (Kcal/kg)		
1	MCL-1	6.7	40.9	11.5	40.9	4693.68	164	82.4
2	MCL-2	9.6	31.7	17	41.7	3733.78	165	88.7
3	SECL-1	5	22.55	28.9	43.55	4586.57	160	93.4
4	SECL-2	3.65	23.4	19.9	53.05	5198.17	167	89.2
5	SECL-3	7.45	18.05	17.5	57	4976.11	157	93.2
6	SECL-4	4.55	23.95	16.95	54.55	5644.47	163	90
7	SECL-5	2.2	31.8	19.6	46.4	5839.6	161	83.7
8	SECL-6	6	19.4	21.25	53.35	5220.5	172	84.9

9	SCCL-1	7.5	22	24.85	45.65	4827.66	168	81.6
10	SCCL-2	6.1	18.9	28.2	46.8	3693.48	165	82.4
11	SCCL-3	7.15	28.55	18.15	46.15	6265.17	149	86.8
12	CCL-1	2.85	20	37.45	39.7	5361.26	146	113.5
13	CCL-2	3.6	20	26.5	49.9	3862.16	170	82.6
14	INDO-1	6	27.6	9.1	57.3	4693.88	190	75
15	INDO-2	5.2	24.7	11.2	58.9	4417.4	146	118

Table 2 Result of Volatile matter, CPT and EMF of 1 day and 3 day oxidized coal samples

Sl No.	Sample	1 Day oxidized coal sample			3 Day oxidized sample	
		VM(%)	CPT	EMF(mv)	VM(%)	EMF(mv)
1	MCL-1	45.5	234	67	53.85	54.7
2	MCL-2	37.3	236	75.9	48.7	58.3
3	SECL-1	28.4	235	78.2	43.9	53.3
4	SECL-2	27.4	243	79.6	42.3	63.5
5	SECL-3	23.15	227	73.6	50.25	50.8
6	SECL-4	29.05	232	64.2	43.05	60
7	SECL-5	35.7	235	76.5	47.2	65.5
8	SECL-6	25.15	235	78.5	37.7	63.2
9	SCCL-1	26	240	66.2	35	56.8
10	SCCL-2	26.3	240	69.2	41.15	46.6
11	SCCL-3	34.25	228	73.9	41.65	49.65
12	CCL-1	24.65	229	89.4	31.25	64.9
13	CCL-2	25.2	248	64.5	34.7	56.3
14	INDO-1	32.65	268	66.1	43.2	51.3
15	INDO-2	29.85	227	93.6	39.9	64.2

From the experimental results it was found that the volatile matter content of the coal increases as the sample is oxidized while the liability of the coal to spontaneous heating decreases with oxidation. For coals with high fixed carbon content the change in liability of spontaneous heating is significant even after 1 day oxidation. While in case of coals with low fixed carbon content the change in liability of spontaneous heating is not too significant. It is expected that finding of this work will help in deciding whether to adopt any precautionary measures for oxidized coals.

REFERENCE

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CHAPTER 1

INTRODUCTION

CHAPTER 1

1. INTRODUCTION

1.1 GENERAL

Fire is associated with coal mining and storage from its beginning. Spontaneous heating is one of the main causes behind such fire. Jharia coal field of India is severely affected by such fire. The first fire was reported here in the year 1916. Now no. of fires has increased upto 70 covering an area of 17 sq km. An estimated 50 million tonnes of good quality coking coal have been lost due to such fire and 200 million tonnes of coal are locked up due to such fire. Along with the loss of coal, these fires also result in severe environmental damages. In case of coal storage some portion of the coal oxidizesduring the extraction operation, loading and transportation. Therefore careful assessment of spontaneous heating liability of both fresh and oxidized coals are required to prevent such instances of fire both in cases of storage and extraction of coal. A lot of research has been done previously to understand the spontaneous combustion process and prevent it.

When coal is exposed to atmosphere, it absorbs oxygen at the external surface as well as in internal surfaces or micro pores. The reaction between coal and oxygen is exothermic with the evolution of heat. This heat produced is dissipated by air surrounding it. If the dissipation rate is less than the accumulation rate then temperature of the coal increases. As the temperature reaches the critical temperature, reaction between coal and oxygen becomes self-propellant. When the temperature reaches the ignition point, coal catches fire.

The liabilityof coal to spontaneous heating is assessed in different countries by different experimental techniques. These techniques are Crossing Point Temperature (CPT) in India, Russian U-index in Russia, Olpinski index in Poland. Along with these some other attempts are designed by researchers all over the world. These attempts include Differential Thermal Analysis (Banerjee and Chakravarty, 1967; Gouws and Wade, 1989), Wet Oxidation Potential analysis (Singh et al., 1985; Tarafdar and Guha, 1989), Gas Indices studies (Panigrahi and Bhattacharjee, 2004; Singh et al., 2007), Critical Air Blast (CAB) rate (Panigrahi and Sahu, 2005) etc.

In present work Crossing Point Temperature (CPT) and Wet Oxidation Potential and Differential thermal analysis (DTA) is used for assessment of spontaneous heating liability of fresh and

oxidized coals. The oxidized coals are obtained from the fresh coal by oxidizing it at 200^0c for 1 day and 3 day respectively.

1.2 OBJECTIVES

In order to assess the spontaneous heating susceptibility of fresh and oxidized coals, the present work has been planned with the following objectives:

- Collection of the various works done by previous researches both national and international on this topic.
- Collection of coal samples from different mines of country.
- Oxidation of coal samples to produce oxidized coal samples.
- Determination of intrinsic property of both fresh and oxidized coals.
- Assessment of spontaneous heating liability of both fresh and oxidized coals by Crossing Point Temperature, Differential thermal analysis and Wet Oxidation Potential.

CHAPTER 2

LITERATURE REVIEW

CHAPTER 2

2.LITERATURE REVIEW

Several research works and studies has been carried out worldwide by different researchers to assess the spontaneous heating liability of coal. Some of these research works has been summarized below:

Tarafdar and Guha (1988) reported results of wet oxidation of coal by alkaline permanganate solution. Seven coal samples were taken. Each coal samples were ground to -212 micron and subjected to wet oxidation using a decinormal solution of potassium permanganate in 1 normal potassium hydroxide solution. Coal was added to the solution. Potential measurements were made between a saturated calomel electrode and carbon electrode immersed in a suspension of .5 gram coal powder in 100 ml of permanganate solution at ambient temperature of 30^0c. A correlation was done between the wet oxidation potential and CPT.

Kucuk et al. (2002) carried out experiments to evaluate the effect of particle size, moisture of coal an humidity of air on the spontaneous heating characteristics of some Turkish lignite. The experimental system contains programmable oven, two stainless steel reactors(30 mm diameter, 140 mm long) furnished with three chromel-alumel thermocouples, an air nitrogen flow system and a multi channel chart recorder to record continuously thermocouple outputs. At lower gas flow rate the air temp.and nitrogen tempincreases in parallel with oven temperature but lower than it as the oxygen available is insufficient to oxidize the coal. Nitrogen temperature is determined to measure the inert temperature but as gas flow rate increases, at nearly 50 ml min^{-1} and 160^0c temperature, the air temperature increases above the oven temperature as the oxygen available becomes sufficient cause oxidation. If the flow temperature increases to 100 ml min^{-1} the air temperature again becomes less than oven temperature due tothe dissipation of heat produced by the air current. Moisture content of the coal is varied by decreasing the size of the coal. As the moisture content increases, the air temperature becomes more than oven temperature but in case the moisture content is too high the temperature of oxidation increases due to evaporative cooling of moisture present in the coal. With the increase in humidity of air the CPT increases as the moistened air has high thermal conductivity and heat capacity compared to

normal air. Similarly with the decrease in size the CPT decreases due to increase in the surface area available.

Smith and Glasser (2002) carried out experiments to investigate the range of coal properties that are critical for heat generation in coal medium. They used temperature-time curves known as thermal characteristics to determine liquid phase kinetics. A semi-adiabatic reactor in a temperature controlled environment, thermocouple, a data logger, gas chromatograph for measuring gas concentration is used in this experiment. Total 9 no. of samples ranging from South African anthracite to Australian lignite are taken and seven runs are done per sample. In the experiment the heater inside the reactor is turned on and the voltage, temperature and current is measured until steady state is reached. After reaching steady state the heater is turned off and decay back to its temperature is monitored. Then the same experiment is carried out in presence of coal. It gives us ideas about heat capacities, heat of reaction and activation energy variation for different coal samples.

Wang et.al (2002) studied the chemical reaction occurring during the low temperature oxidation of coal. Coal oxidation occurs at both at the external surface of coal particles and internal surface of pores. The rate of oxidation corresponds to rate of oxygen consumption. It is determined from the isothermal flow reactor. Two parallel reaction occur during coal oxidation at low temperature. One is chemisorption of O_2 on surfaces of coal pores and the formation of unstable carbon oxygen complexes. The chemisorption process consists of formation of peroxide as a result of coupling of bi-radical oxygen molecule (-o-o-). The evidence for it is the change in free radicals of coal sample after the first contact of coal sample with the oxygen. Decarboxylation reaction also occurs in the oxidation process. Kinetic modeling is done by them to describe the oxygen adsorption or consumption process.

Lu et. al (2004) carried out experiment to find the gases that can predict the spontaneous combustion of coal in the low temperature range. The oxidation of a coal particle depends primarily on the size, porosity and reactivity of the coal particle. The reaction regime is also controlled by molecular diffusion for large-sized coal particles but in case of very fine particles (less than 50–500 micro m) the reaction becomes kinetically controlled. The heater reactor used for this experiment mainly consists of four sections, the oxidation section comprising a coal sample jar, heater and at temperature oven; coal temperature monitor and control unit; the

ventilation system comprising of a fan, tube, control valve and flow meter; the gas component analyzing system. Bituminous coal for experiment was obtained from Longgu colliery in the north of Jiangshu province China. 5 no. of coal samples were taken. A coal sample (1–30 mm, 1000 g) uncovered recently was placed in a heater reactor. The first gas collection temperature was 30^{o}C, and gas samples were collected at intervals of 10 ^{o}C. The air flow is from the bottom of the apparatus and the coal is heated simultaneously till the temperature reaches a predetermined value. This state is maintained for 2 minutes. The gas sample is taken. The experiment was ended after reaching a temperature of 200^{o} C. It was seen that CO was detected from 49.8^{0} C to 54.9^{0} C and ethylene from 64.3 to 75.3^{o} C. From the results it is concluded that when CO gas was detected in the airflow of the coal workface the temperature of coal in the gob area reached 50 ^{0}C, if CO and C_4H_4 are detected simultaneously, the temperature of the coal had reached 64 ^{0} C, and if the concentration of CO and C_4H_4 increased continuously, it indicate that coal self-heating was developing.

Beamish and Arisoy (2006) conducted self-heating test on a sub-bituminous coal sample containing mineral matter in a range from 11.2%-71.1%. The samples used in this study were firstly wrapped in plastic cling wrap and then aluminium foil and finally an outer layer of masking tape. They were stored in freezer until adiabatic experiments are carried out. The coal was crushed to -212 micron just before the experiment to minimize the effect of oxidation on fresh surfaces. A 150 gram of crushed coal sample dried for 16 hours under nitrogen at 110^{0}c then cooled to 40^{0}c. After the stabilization of sample temperature, the oven tracked the coal temperature rise due to oxidation. The heat produced due to the oxidization of mineral matter free coal is absorbed by both mineral matter and mineral matter free coal respectively. The oven was automatically setup to cut the oxygen supply after reaching 160^{0}c. A graph is plotted between the temperature increase rate versus temperature. A third degree polynomial is fitted into the graph between ln(dT/dt) and 1/T. Another adiabatic oven experiment was carried out in a known coal and the coefficients were determined. Then both these functions can be correlated such that they give their temperature increase tendency for different mineral matter content both theoretically and analytically. It is seen that with the increase in mineral matter content the self-heating tendency of coal decreases.

Zhang and Kuenzer(2006) carried out experiments to measure thermal characterstics of coal fire measured insitu. Coal fire surface anomaly temperatures and surface temperatures for typical

background materials (unaffected by coal fires) were measured using a Raytec thermometer and contact thermometers. The Raytec thermometer allowed for highly precise radiant temperature measurements without being in direct contact with the surface. Precision is 0.1 °C of readings up to 900 °C in the 8 μm to 14 μmdomain. The contact thermometer has a handle and a 12.8 cm long probe looking like a screwdriver. It measures the kinetic temperature at the head of the probe. Measurements were performed along horizontal profiles. Measuring points were clearly defined with 10 cm, 1 m or 10 m distance depending on profile length and measurements over the diurnal cycle were always repeated at exactly the same points. Vertical profiles sub-surface or inside of cracks were performed at centimeter range measuring intervals. Radiant temperatures on a sparsely vegetated light sand dune can vary for around 28 °C between sunrise and sunset. In a coal dump pile, representing a dark surface, temperatures for a slope facing Southeast were 20 °C higher than that for a slope facing North between 10:00 to 14:00. These temperature differences (variation) caused by uneven solar heating due to different albedo, slope, aspect and vegetation cover can mask thermal anomalies related to coal fires. This is especially important when trying to detect coal fire related thermal signals in airborne or space borne remotely sensed imagery, or when mapping coal fires in-situ. Night-time and especially pre-dawn data (or mapping) would thus be most suitable for fire mapping and to distinguish the fires against their background.

Denis et.al (2007) studied the spontaneous heating of coal in witbank and Sasolburg coal fields in South Africa. The witbank coal fields are hosted by Vryheid formation. These collieries have used bord and pillar method mining with typical low recovery ratios. During reopening of old workings ingress of air into galleries resulted in chemisorption and oxidation leading to spontaneous combustion. Eight gas samples were collected from the witbank coal field and five from Sasolburg coal field using a hand operated pump with a Teflon inlet and exhaust tube. Each samples were studied by X ray powder diffraction in combination with scanning electron microscopy. The analysis showed that condensation byproducts were mostly sulphur and sulphur bearing mineral. Gases emitted in coal fire consisted of four main components i.e. aromatic compounds (benzene, toluene), aliphatic hydrocarbons, halogenated hydrocarbons, greenhouse and other gases.

Fei et.al (2008) compared thespontaneous combustion of two victorian brown coal and a Pakistan lignite with the products of same coals which have been dried by mechanical thermal

expression technique. It is carried out at 125^0c and 200^0c under pressure of 5,10 and 15 mpa. The coal sample (100gm) was loaded into a specially designed filtration cell along with an equal amount of distilled water to produce trapped air free slurry. A piston is inserted and lowered till the top of the slurry. Then the coal was heated to selected temperature by means of the heating jacket surrounding it. The cell is allowed to stay at selected temperature and pressure for 5 minute and 20 minute respectively after which it is allowed to cool down. The pellets thus formed along with raw coal and briquette were served to three size fractions x<.5, .5<x<1.4 and 1.4<x<2.8 mm. Samples were placed in a cube shaped sample holder of stainless steel mesh. A constant packing density is maintained. Three types K thermocouples were placed in the sample holder and fourth was used to measure the temperature of the oven. It is heated at 8^0c per minute until the temperature reaches a maximum value. The temperature of oven and sample are measured using a data logging device. The minimum crossing point temperature required to make thermal runaway to occur is called T_{crit}. They observed that with increase of particle size and decrease of moisture content T_{crit} starts to increase.

Sahu et.al (2011) applied principal component analysis (PCA) using moisture, volatile matter, ash and crossing point temperature as the parameters to classify the coal seams into three different categories. Samples were collected from all the subsidiaries of CIL except NECL. The rank of these coals varie from high volatile sub bituminous coals to anthracite. Moisture, volatile matter and ash were determined as per IS 1350 part 1. CPT was determined as per the setup described by Panigrahi et al. (1996). The variables were normalized by dividing the selected variables by their maximum value so that all data varied from 0 to 1. The correlation matrix was obtained in the form of Euclidean distance matrix. Euclidean distance indicated the intensity of dissimilarity between two coal samples. Considering M, VM, A and CPT as variables, and applying the PCA methodology, the corresponding eigenvalues and eigenvectors for the Euclidean matrix were calculated using SYSTAT version 7.0 software. All the samples having close PCA values are clustered into one group. Each cluster represented sample with nearly same susceptibility so by knowing the susceptibility of some sample of that cluster the liability of the whole cluster can be known.

Xie et.al (2010) had developed an ethylene enriching system based on its physical adsorption and desorption properties to increase detection sensitivity of ethylene concentration in mine air by about 10 times. The ethylene enriching system (EES) consists of a gas flow system, an

ethylene enriching and adsorption system, a temperature controlled and a gas detection system. First a gas cylinder containg a known ethylene concentration is connected to the adsorption chamber through the inlet valve. The gas cylinder and inlet valve are then opened allowing the gas to follow into the chamber at 15 ml/minute and the outlet valve is kept open. The gas flowing out of the outlet valve is sampled at 30 min interval and then ethylene concentration is measured with a gas chromatograph. When EES value reached 15 ppm the fire controlling gel and spray are applied.

Xuyao et.al (2010) carried out experiments for investigating the crossing point temperature (CPT) of different ranks of coal. The temperature at point where the coal temperature begin to exceed the surrounding temperature is the CPT of the coal. In the experiment 50 gram of coal sample were packed into the coal reaction vessel. A thin asbestos of was laid on the top of the coal sample to prevent the gas tube from being plugged. The programmed temperature enclosure was set to run at a constant temperature of 40^0c while dry air with an oxygen concentration of 20.96% was permitted to flow through the coal reaction vessel at a rate of 8ml/minute. The temperature logger was used continuously to measure the coal and surrounding temperatures. The temperature was increased at a rate of $.8^0$c/minute. When the coal temperature reached 70^0c, the flow rate of dry air was changed to 96ml/minute. The experiment was completed when the coal temperature was 5^0c higher than the surrounding temperature. It was seen that coal samples with low moisture content show smooth trend curves and with high moisture content show a delay in the rise of temperature.

Kim and Sohn (2011) studied numerically the spontaneous ignition of coal stock piles and its suppression in a coal storage. The reactive flow field in a coal stockpile is simulated by solving the continuity, energy, momentum simultaneously. The dominant source for coal heating was the coal oxidation. First analysis was done for high rank coal with low moisture content. The rate constant was taken in the arhenius equation form. The temperature exponent in the arhenius equation is so adjusted that the spontaneous ignition time predicted by the numerical analysis agrees well with the experimental data. At far away from the stock pile free flow conditions were present. The oxidation process is simulated by a transient analysis with a time gap of 1 day i.e. 86400sec. Based on the new idea obtained from the numerical analysis suppression ideas can be devised newly.

Liang etal. (2011) carried out research to assess the inhibiting effect of sand suspended colloid in suppressing the spontaneous combustion of coal-sand suspended colloid is composed of inorganic mineral gel, organic polymer and the dispersant for which the inorganic gel is the basis. The inorganic materials in the sand-suspended colloid cannot absolutely dissolve in water but soak water and swell. After mixing the colloid can disperse into particulates which generally have electronegative surface. The organic polymer is a kind of amylase polymer distilled from savageness plant of alga, which solution could link with the divalent ions in the inorganic mineral gel, such as Ca^{2+}, Cu^{2+} and Pb^{2+}, to form an extensive space reticulation structure. The fresh coal samples were chosen from Huxi coal mine of Jining in Shandong province and Yuanzhuang mine of Huaibei in Anhui province. The samples were crushed into coal particles of 40–80 mesh with 160 g of weight, and then the coal particles are dehydrated in the vacuum oven with drying temperature of 25 C. The sample was divided into four parts with 40 g respectively. Calcium chloride, soluble glass and sand-suspended colloid were selected as the inhibition substance samples. 40 g of the original coal sample was weighed and put into the paper cup, and mixed with 4 g of inhibition substance solution, then the mixed coal samples is dehydrated in the laboratory under the room temperature. Then the mixture is enclosed in the coal pot at once. Eight coal samples were put into the coal pot respectively for experiments. The inhibition effect of three inhibition substances on the acceleration oxidation of each coal sample is found out to be different. For the lignite, the order is given by sand-suspended colloid > soluble glass > calcium chloride. For the fat-gas coal it is given by soluble glass > calcium chloride > the sand-suspended colloid.

Choi etal. (2011) investigated the tendency of re-adsorption, low temperature oxidation and spontaneous combustion of an upgraded low rank coal. The raw coal was upgraded by grinding and sieving it to obtain 0.5–1.3 mm particle size. It was made slurry by mixing with kerosene solvent. Up to 4.0 wt% of asphalt, as a heavy oil additive, was added to the slurry. The weight ratio of the solution (kerosene+asphalt) and the raw coal was adjusted to 1:1. The slurry was retained at 140 °C for 30 min to evaporate the moisture in the coal. The pressure of the reaction vessel varied in the 0.1–0.3 MPa range. After the evaporation the solid coal was filtered and dried in an oven at 130 °C to remove the remaining solvent. Raw coal and upgraded coal samples were dried again at 105 °C for 5 h in nitrogen to remove the moisture that might have been adsorbed while the sample was being preserved. Two vessels containing coal samples (35 g

each) were installed in a programmable oven. The oven temperature was set at 40 °C under nitrogen. After the oven and vessel temperatures were brought to equilibrium, the oven was heated at the rate of 0.5 °C/min while supplying air to the one and nitrogen to the other vessel at a rate of 30 mL/min. The coal receiving nitrogen served as a reference. The temperatures of the oven and the two samples were constantly monitored. When the amount of asphalt reached 0.5%, the CPTs increased a little compared to cases with no added asphalt, and when the amount of asphalt reached 1.0%, the CPTs increased considerably. This seemed to occur because the asphalt coated the coal surfaces during the upgrading processes preventing the functional groups on the coal from contacting oxygen. However, when the amount of asphalt reached more than 1.0%, it showed no further effect on the increase of the CPT. These results indicate that a certain amount of added asphalt during the upgrading process will improve the CPT.

Yuan and Smith (2011) studied the effect of ventilation on the spontaneous heating of coal in an underground coal mine. They crushed the coal sample to the 100 -200 mesh size fraction and dried in the oven with a nitrogen flow rate of 200 cm^3/min at 67 ^oC prior to the experiment. 150 g of coal sample is used in each experiment. A programmable isothermal oven whose temperature can be held constant from 40^o c to 200^o c within an accuracy of 0.5^o c was taken. It takes about 30 min to reach the isothermal condition in the oven. A stainless steel container containing a 6cm dia by 10 cm high brass wire masket along with the coal. Three K type thermocouples of 0.5 mm dia. 3 cm above and below the coal samples were placed to measure the coal temperatures. Nitrogen gas was introduced through ports in the bottom and exits through the top of the container. When the coal sample reaches the oven temperature, the nitrogen flow is switched to the same flow rate of dry air. The exhaust gas exiting the container passes through a water trap before going through oxygen, carbon monoxide (CO), and carbon oxide (CO_2) gas analyzers. The CO and CO_2 gas concentrations are measured continuously using an infrared gas analyzer, while the oxygen concentration is monitored continuously by a paramagnetic oxygen analyzer. Tests are terminated when the coal temperature begins to decrease or the temperature reaches 250 ^{0} C. From the temperature-time and exit oxygen concentration curve of coal sample the bottom coal temperature exhibited a clear three stage increase. The first stage is a slow increase, the second one is a fast increase, and the third one is a slow increase again. The top temperature was much lower than the center and bottom temperatures. The center temperature increased slightly faster than the bottom in the first stage,

while the bottom temperature increased faster than the center in the second stages, probably because not enough oxygen was available at the center.

Jianfanget.al(2012) carried out experiments to show the relationship between oxygen consumption rate and heating to know when the critical temperature of the coal is reached. Coal samples were collected from 2, 4 and 6 no. coal seams of the Xuecun Mine of Fengfeng Group respectively with the sampling method of notch grooving. Before sampling, the oxide layer on the surface of the coal seam is removed. Four beelines from the roof to the floor are marked on the face, perpendicular to the roof to the floor. The interval between lines was 0.15 m and having a depth of 0.05 m. A piece of plastic cloth was placed on the floor at each sampling point to let the picked coals fall on it. The coal was reduced to about 1 kg for each sample with the shrinkage method of coning. The coal samples were filled into separate thick sealed bags and then it was sent to laboratory. 5 g coal sample was selected of size 80–100 meshes into heating case. Compressed air was injected in it at a speed of 80 ml/min, and the speed of heating is at 1 C/min. The constant temperatures set by programs are 30^0C, 45^0C, 60^0C , 75^0C, 90^0C, 120^0C, 150^0C, 156^0C, 180^0C; if the heating temperature has reached any one of them, the temperature will be maintained for a period of time and at the same time, the gas chromatograph begins to take in gases to do chromatographic analysis of gas components and corresponding concentration. After the temperature has been maintained for a certain period of time, heating starts again until the next constant temperature is reached, then keep the temperature and take in gases to do chromatographic analysis. During the process of heating-up and oxidation, the oxygen consumption rate steadily increases with temperatures of coal samples. When temperature reaches to a certain value (around 150 C), the oxygen consumption rate increases sharply. Moreover, the increasing trends of these three coal samples are completely same. The temperature at which the oxygen consumption rate begins to increase sharply is called the critical temperature.

CHAPTER 3

THEORIES AND MECHANISM OF SPONTANEOUS HEATING

CHAPTER - 3

3. THEORIES AND MECHANISM OF SPONTANEOUS HEATING

Spontaneous combustionoccurs by self-heating i.e. increase in temperature due to exothermic internal reactions followed by thermal runaway i.e. auto oxidation which rapidly accelerates the temperature and finally ignites the coal. Coal fires require three basic elements given below.

- Oxygen
- Heat
- Fuel

When coal is exposed to air a gradual absorption of oxygen occurs and the temperature rises. Usually in such a case the heat is conducted to the adjacent layer of coal or rock. Then it is dissipated and cooled by the air. However, if the coal is fresh and there is not an adequate current of air to cool the generated heat, the heat is accumulated and the temperature rises. If the temperature rises to a point above critical temperature then auto-oxidation of coal occurs leading to spontaneous combustion but if it is less than the critical temperature then no spontaneous combustion occurs instead weathering takes place. Weathering results in chemical and physical degradation of coal. Calorific value and caking property of coal deteriorate with weathering.

3.1 THEORIES OF SPONTANEOUS HEATING

3.1.1 Pyrite theory

In pyrite mines heating due to oxidation of pyrites are very common process. Iron disulfide is associated with coal in two form, i.e. cubic yellow pyrite and rhombic marcasite. Marcasite has been reputed to be more reactive towards oxygen than cubic yellow pyrites. Li and Parr (1926) found that both form oxidized about the same rate.

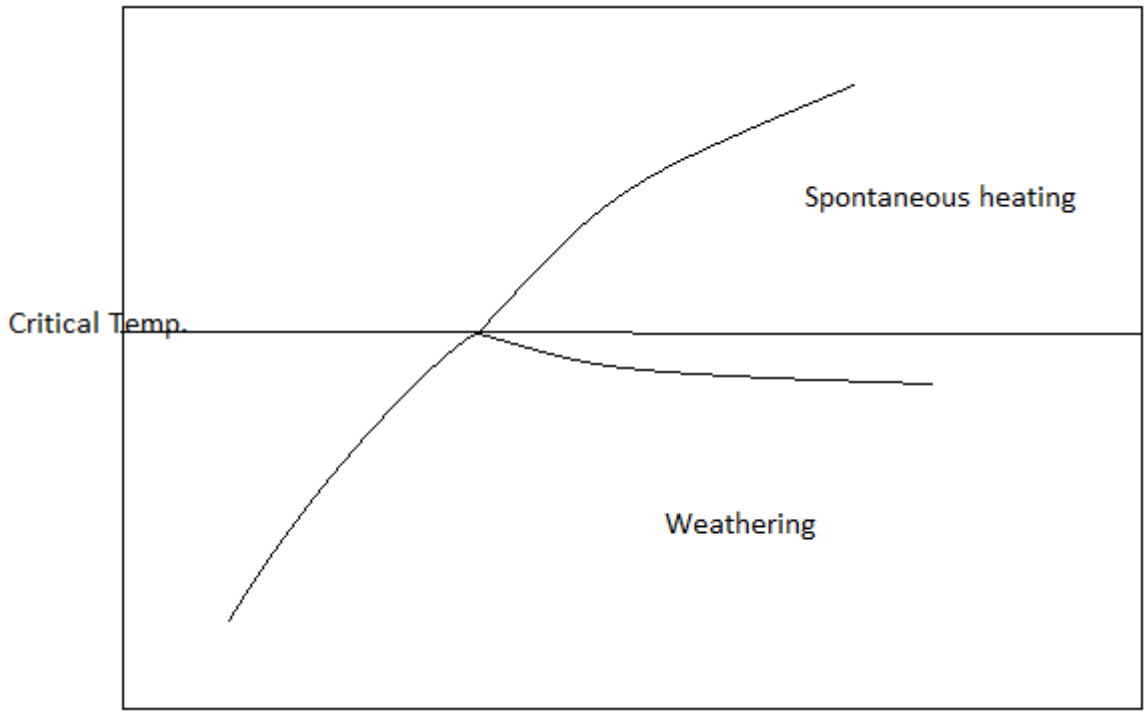


Figure 3.1 Figure showing weathering and spontaneous heating

Heating of coal could be caused by oxidation of iron pyrites and in finely powdered and dispersed state in the presence of moisture. The heat of oxidation of pyrites was determined by Lamplough and Hill (1912-13) who found that a mean value of 13.8 J per mL of oxygen consumed. The reaction of iron pyrites with oxygen and moisture gives products of larger volume than the original pyrite hence opening more pore area of exposure for oxygen, which is an exothermic reaction.

$$2Fe\ S_2 + 7 0_2 + 16 H_2O = 2\ H_2SO_4 + 2\ Fe\ S0_4.7\ H_2O + 316\ kcal$$

The above equation is an exothermic reaction. It also yields reaction product having greater volume than the actual pyrite, with the result that would break open any coal and thus exposing a greater surface of coal to the air. It is also observed that sulphonated coal have greater reactivity towards oxygen, especially in the presence of iron oxide. It might have an considerable effect if its concentration in finely dispersed from exceed 5 to 10 %. If the pyrite present is less than 5%, then its effect is negligible. However, it has since been shown that coals that do not contain

pyrites are also prone to spontaneous combustion. The presence of pyrites is merely a contributory factor.

3.1.2 Bacterial action

Heating in hay stacks and woods occur due to bacterial action. Earlier it was thought that spontaneous heating in coal was also due to bacterial action. Further research revealed that bacterial action could help in heating but it don't have significant role in spontaneous heating.

Coward showed that bacteria can live on coal and these bacteria in some cases can increase the temperature of coal.

Graham (1914 -15) found that sterilized coal oxidized at the same rate of as the unsterilized coal, and concluded that the mechanism of oxidation did not include bacterial activity. A similar conclusion was drawn by **Winmill** (1914-15)

Fuchs (1927) showed that bacteria can only cause slight heating but it don't have significant role in spontaneous heating.

3.1.3 Humidity theory

Scott (1956) has stressed the importance of humidity in relationship to spontaneous combustion. By means of a specially design micro calorimeter, he was able to show that the quantity of heat liberated by atmospheric oxidation of coal was much less than the quantity required to remove water from coal. He concluded that if evaporation of water can be induced at the heat of a heating, then the temperature of the heating would decrease.

Mukherjee and Lahiri proposed a mechanism of the reaction between water and coal at $100^{o}C$. It also explains other possible sources of CO and CO_2

$H_2O \rightarrow (H_2) + (O)$

$C + O \rightarrow CO$

$(CO) + (O) \rightarrow CO_2$

3.1.4 Heating due to earth movement

Crushing of coal under pressure of super incumbent rock can be a source of heating contributing to spontaneous combustion. Sudden roof fall and resultant adiabatic compression may also elevate ambient temperature. But it has been observed that though the heat from earth movement would contribute to spontaneous combustion, the oxidation of coal is most important factor. Crushing of coal increases surface area of coal for oxidation making it more potential to auto oxidation.

3.2 MECHANISM OF SPONTANEOUS HEATING

Research has concluded that natural coal oxidation obeys an Arrhenius-type rate law. The relationship between reaction chemistry, rate of reaction constant and temperature determines the Arrhenius equation .

$\ln(k) = \ln(A) - E_A/(RT)$

k = Rate of reaction constant

A = Frequency factor

E_A = Activation energy

R = Gas constant

T = absolute temperature

The dependence of the activation energy on the temperature was observed. In the range between 23 and 70°C it was estimated to be 47.1 kJ/mole following first order kinetics. Higher activation energy of 82.0 kJ/mol was obtained in the range between 70 and 90°C. Below 70°C the chemisorption is dominant while above this temperature peroxide decomposition and formation of oxygen functional groups on the coal surface is dominant. Chemisorption of oxygen on the coal surface follows the reaction:

$C_{100}H_{74}O_{11} + 17½\ O_2 \rightarrow C_{100}H_{74}O_{46} + 2.5$ MJ/kmol

Observation of free radical concentration shows that the free radical decay obeys secondorder kinetics with these potential reactions:

$R + O2 \rightarrow ROO$

$ROO^{\bullet} + R_1H \rightarrow ROOH + R1^{\bullet}$

$ROOH \rightarrow RO^{\bullet} + {}^{\bullet}OH$ Chain breaking

$RO^{\bullet} + R_1^{\bullet} \rightarrow ROR_1$ Second order radical termination

The rate of oxidation increases by a factor of 2.2 for each 10°C rise in. A more specific analysis yields that the rate of oxidation is slow below 40°C but accelerates by a factor of 1.8 thereafter, also the critical temperature above which the oxidation and self-heating process becomes self-sustaining is about 50°C for lignites and 70 – 80°C for bituminous coals. In general, the oxidation increases at ten times its usual rate as the temperature rises from 30 – 100°C. In contrast the rate of oxidation slows as oxidation progresses. The reason for this is that the initial surface oxidation of the coal particles results in the formation of oxidized material (“oxycoal” compounds) producing a barrier. Oxygen must penetrate through this formed barrier for further

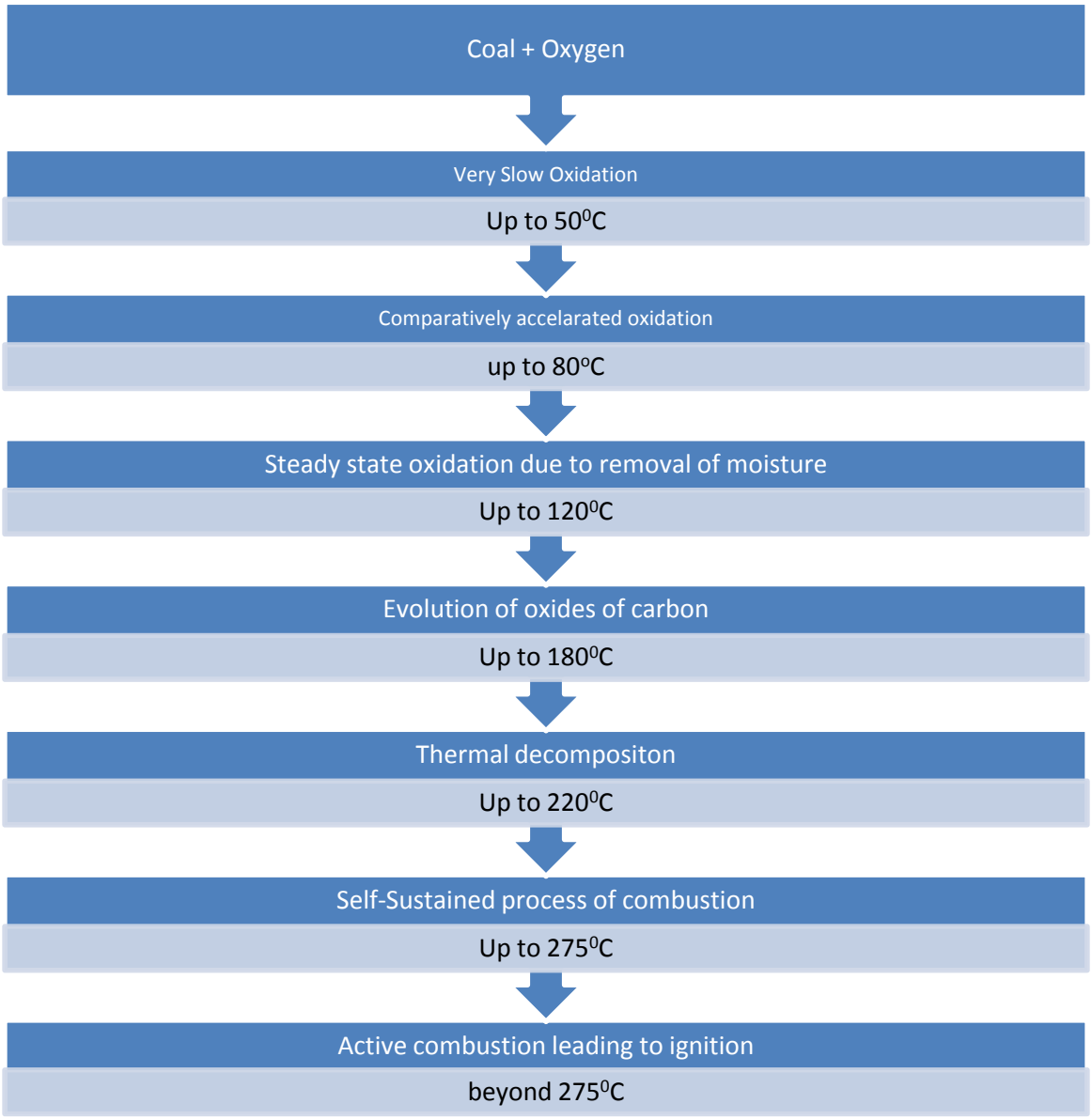
reaction to proceed. It becomes physically more difficult for oxygen to penetrate deeper into the coal, thus slowing the reaction to a minimum rate. At a constant temperature the rate of oxidation slows by one-tenth of its value per hourup to 100 hours from onset.

Three types of processes are involved in the coal oxidation. Oxygen is physically and chemically adsorbed by the coal. Physically adsorbed oxygen (E_A=42 kJ/mol) diminishes in quantity with gradual temperature rise and becomes inappreciable beyond 50°C. Chemical adsorption (E_A=139 kJ/mol) leads to the formation of coal-oxygen complexes and oxygenated carbon compounds (i.e. carboxyl and carbonyl groups). The formation of the carbon compounds proceeds selectively. The benzylic positions, being the most reactive, are first oxidized to produce carbonyl groups and carboxylic acids. The aliphatic groups of coal are oxidized to form aldehydes, and then are oxidized further to form carboxylic groups and esters. Finally, this results in the formation (E_A=105 kJ/mol) and release of gaseous products, typically carbon monoxide, carbon dioxide and water vapor.

From the low temperature formation of coal-oxygen complexes to the outbreak of spontaneous combustion, the coal heats at an increasing rate. This heat is dependent on whether enough air is available and enough heat is retained to sustain the reaction. At temperatures above 80°C the coal-oxygen complex breaks down and releases greater amounts of heat with the reaction:

Coal oxygen complex -> $CO + CO_2 + H_2O$ + heat

The decomposition of the chemisorption intermediates mainly generates CO while the liberation of CO_2 comes from the decomposition of the carboxyl groups. Three simultaneous reactions proceed in the thermal decomposition process. The carbonyl groups are supposed to be generated via a dehydroxylation process. The decarboxylation results in gaseous products, including CO_2, CO and H_2O, and the decarbonylation process liberates the same species. At higher temperatures of about 150°C decomposition of carboxyl and carbonyl groups occurs, leading to the production of carbon dioxide and carbon monoxide along two independent reaction pathways. In addition to this processes, the reaction involved in oxidation of hydrogen in coal to form water is also exothermic. This is of particular importance for lowrankcoals that have high inherent hydrogen content.



Stages in spontaneous heating of coal

3.3 FACTORS AFFECTING SPONTANEOUS HEATING OF COAL

Aside from the natural affinity of coal to self-heating, a number of factors are significant when determining the risk of spontaneous combustion and are presented below

3.3.1 Coal rank

Spontaneous combustion is a rank related phenomenon. The tendency of coal toself-heating decreases as the rank increases, with lignite and sub-bituminous coalsbeing more susceptible to self-heating than bituminous coals and anthracite. Asrank decreases, inherent moisture, volatile matter and oxygen and hydrogen contentsincrease. Medium to high volatile coals with a volatile matter content higher than18% wtdaf basis have a faster oxidation rate than low volatile coals and are thereforemore prone to spontaneous combustion than low volatile ones. Furthermore lowerrank coals often have a greater porosity than higher rank coal and therefore moresurface area is available for oxidation. Low rank coals also contain long chainhydrocarbons making them less stable than high rank coals which have a lowerhydrocarbon component. However, the oxidation rate for coals of the same rank may vary within a wide range.

3.3.2 Petrographic composition

The petrographic composition of a coal is determined by the nature of the original plant material from which it was formed and the environment in which it was deposited rather than the degree of coalification (i.e. rank). The homogenous microscopic constituents are called maceralsand can be distinguished in three groups. Vitrinite consists of woody material while exinite consists of spores, resins and cuticles and inertinite, the third maceral group, consists of oxidized plant material. For samr rank coal, as the inertinite content of a coal increases, its self-heating propensity decreases. The general trend also indicates an increase in self-heating propensity with either increasing vitrinite or liptinite content.

Liptinite>Vitrinite>>Inertinite

Coal ranks seem to play a more significant role in self-heating than the petrographic composition.

3.3.3 Methane content

The emission of methane from coal forms a sluggish atmosphere and may inhibit low temperature oxidation mainly in coals with high contents of gas but methane also posseses the potential as a source of energy. Furthermore, as the methane desorption decreases sharply with

time, more of the coal surface will be exposed to oxidation. Coals with methane gas emission index less than 5 m^3/t show high oxidation rates while coals containing above 8 m^3 of methane/t show low oxidation rates.

3.3.4 Minerals

Many chemicals in mineral form affect the oxidation rate to some extent, either accelerating or inhibiting it. Alkalies may have an influence of acceleration while borates and calcium chloride can act as retardants. The oxidation process is also promoted if ankerite, a constituent of the coal mineral matter, is present. In contrast to this silicia and alumina retard the oxidation. Pyrite (FeS_2) evolves heat from aerial oxidation and was believed to be the cause of the spontaneous heating of coal. The heat generation locally promotes the self-heating process of coal but the reaction products have a greater volume than the original pyrite, with the result of breaking open any coal in which they are embedded and thus exposing a greater surface of coal to the air.

3.3.5 Moisture

The effect of moisture on the self-ignition is twofold: on one hand the vaporization of moisture consumes energy and hence the ignition process is impeded. On the other hand a promotion of self-ignition by the wetting of materials has been observed.

dry coal + moisture → wet coal + heat

In addition to the heat of wetting moisture simply blocks the access of oxygen through the coal pores. The water vapour diffusing outwards through the pores reduces the oxygen partial pressure and hence lowers the rate of the reaction.

3.3.6Particle size and surface area

Oxidation increases with increasing fineness of coal and the rate of oxidation of coal with oxygen of air is proportional to the specific internal surface. The proportional coefficient at low temperatures is small but analysis also show that both rate and extent of oxidation increase with the decrease in particle size, until a critical particle diameter is reached, below which the rate remains fairly constant. A comparison with coal particles of various sizes displays that the smaller particles seem to have a lower alkane content than the larger particles. This can be explained as a decarboxylation reaction and in terms of “micropore” and “macropore” oxidation. Macropore oxidation is particle size dependent. In micropore oxidation the coal particle is

"open" and oxidation is not limited but it is rather controlled by the diffusion of the surface area. For very fine particles the reaction becomes Knudsen diffusion controlled for active coal or kinetically controlled for less active coal.

Oxidation alters the cumulative pore volume of the coal. The cumulative volume of pores greater than 10 nm (100 Ångstroms) radius is larger in the oxidized coal than in the original sample. The reverse case occurs for smaller pores. The surface area of the oxidized coal decreases by about 40% compared to the original coal. A possible cause for this may be that the smaller pores are blocked by adsorbed oxygen and reaction products at the early stages of oxidation.

3.3.7 Physical properties

A number of physical properties such as porosity, permeability, hardness, thermal conductivity and specific heat can influence the rate of oxidation and thus aid in spontaneous combustion. Hardness affects the friability hence the surface area. The rate of heat transportation from the coal depends on its thermal conductivity. Coals with low thermal conductivity tend more frequent to spontaneous combustion.

3.3.8 Friability

The rate of coal oxidation has a linear relation with the surface area of coal. Therefore, the more friable the coal, the greater the surface area exposed to oxidation, thus giving off more heat per unit volume of coal.

3.3.9 Oxygen fraction

It doesn't need any explanation that the volume fraction of oxygen in the gas plays a key role for the rate of the reaction of oxidation. Limiting the supply of oxygen to the active surfaces of the solid matter abates the reaction considerably.

3.3.10 Heat and mass transfer

The transport of the reactants and transport of heat play an important role in the spontaneous heating of coal. Heat is transported away from the sites where it is generated due to temperature gradients. The responsible mechanisms are conduction and convection. Oxygen and water participate in the heat generation processes and are transported by diffusion and convection. Convection may be caused by differences in pressure at the surface (forced convection) and differences in temperature between the surface and the surrounding air (free convection).

3.3.11 Volume to surface ratio

The volume to surface ratio (V/A) is the principal geometrical quantity to assess the self-ignition behavior of solid bulk material, e.g. stockpiled coal. Samples of different shapes, but the same V/A ratio doesn't have the same self-ignition temperature.

3.3.12 Emissions from self-heating and spontaneous combustion

Emissions from coal oxidation, self-heating and spontaneous combustion follow clearly defined patterns. Research has suggested that patterns of gas emissions are not the same for coals from different geographical locations.

CHAPTER 4

EXPERIMENTAL INVESTIGATION

CHAPTER 4

4. EXPERIMENTAL INVESTIGATION

Total 15 no. of samples were collected throughout the country for the purpose of assessment of spontaneous heating susceptibility. These samples were collected from South Eastern Coalfields Ltd. (SECL), Mahanadi Coalfields Ltd. (MCL), Singarenicollieries company Ltd. (SCCL)and Central Coalfields Ltd. (CCL) through channel sampling method. Also samples were obtained from Indonesia to analyze its characteristics with Indian coal. Oxidized coal sample was obtained by oxidizing the coal at 200^0c for 24 and 72 hours. Proximate analysis was done on the coal sample for determination of ash, moisture, volatile matter and fixed carbon percentages. Calorific value of the coal sample was determined by bomb calorimeter. Spontaneous heating susceptibility is determined by Crossing Point Temperature (CPT), Differential thermal temperature (DTA) and Wet Oxidation Potential methods.

4.1 SAMPLE COLLECTION AND PREPARATION

Coal is a highly heterogeneous substance in terms of the inorganic and organic constituents and exhibits wide variability with respect to size and chemical composition of the particles. An estimation of the true value of the desired parameters of a bulk material, to a certain of degree confidence, through analysis on a few grams of test sample is a daunting problem. The basic purpose of collecting and preparing a sample of coal is to provide a test sample which when analyzed will provide the test results representative of the lot sampled. Sampling can be done by several methods

- Channel sampling
- Chip sampling
- Grab sampling
- Bulk sampling

For the project work, sample are collected by channel sampling method as per Indian Standard 436 Part I/Section 1.

4.1.1 Channel sampling

Channel sample consists of cuttings collected from a groove cut in to the rock about 30 cm wide and 10cm deep. Various tools ranging from pneumatic chisel to hammer can be used to cut the sample. Accessibility and rock hardness determine the applicable sampling tool.

Before attempting to take a sample, the rock surface was cleaned thoroughly to remove dust and soluble salts. The method of cleaning depends on the amount of mine dust accumulated on the surface or the degree of alteration of rock surface. Typically cleaning is done by a wire brush, water or chipping a fresh surface.

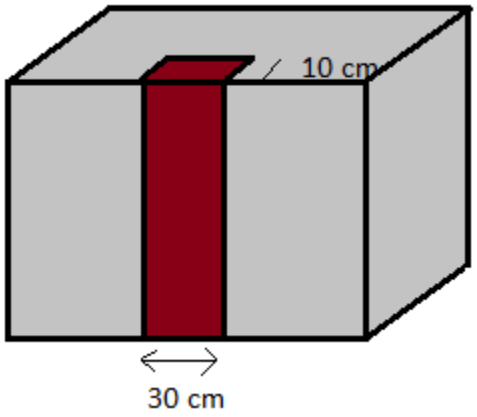


Figure 4.1 Channel Sampling

Then the sample was marked on the prepared surface. This was done by marking two parallel lines on the ore body 30 cm apart at right angles to the bedding planes by chalk. After determination of sample outline the sample is chiseled out. Sample was cut up to a depth of 10 cm. The rock fragments were caught on a canvas tarpaulin on the floor. The collected samples were marked with an identifying label or tag.

4.1.2 Sample preparation

Samples collected from different collieries were brought into the laboratory. The representative of an entire coal sample was obtained by coning and quartering method. Then the coal sample was crushed and passed through an -212 micron sieve. After that the samples obtained were divided in three parts. The first part was kept in an oven at 200^0c for 24 hours. After 24 hours the sample was taken out and kept in a desiccator for cooling. The cooled sample was kept in airtight polythene for further analysis. Then the second part was kept in an oven at 200^0c for about 72 hours. Then the sample was taken out and cooled. After cooling the sample was kept in airtight

polythene. The third part was kept in the air tight polythene without any oxidation for further analysis.

All 45 samples were prepared by the above procedure. Of these 18samples were from SECL, 6samples were from MCL, 6 samples were from Indonesia, 6 samples were from CCL, 9 samples were from SCCL.

Table 4.1 Different samples collected for investigation

Sl no.	Sample name
1	MCL 1
2	MCL 2
3	SECL 1
4	SECL 2
5	SECL 3
6	SECL 4
7	SECL 5
8	SECL 6
9	SCCL 1
10	SCCL 2
11	SCCL 3
12	CCL 1
13	CCL 2
14	INDO 1
15	INDO 2

4.2 INTRINSIC PROPERTY DETERMINATION

4.2.1 Proximate analysis

Proximate analysis indicates the percentage by weight of the Fixed Carbon, Volatiles, Ash, and Moisture Content in coal. The amounts of fixed carbon and volatile matter directly contribute to the heating value of coal. Fixed carbon acts as a main heat generator during burning. High volatile matter content indicates easy ignition of fuel. The ash content is important in the design of the furnace grate, combustion volume, pollution control equipment and ash handling

systemofa furnace.These variables are calculated in an air dried basis. This basis neglects the presence of moisture other than inherent moisture.

4.2.1.1 Moisture content

Coal is hygroscopic in nature. It may absorb or lose moisture depending on the humidity and temperature to which they are exposed. The moisture present in the coal is divided in two categories. One is external while the other is inherent. External moisture can be removed by exposing the coal to the sunlight. After that inherent moisture can be determined as per air dried basis.

Moisture may occur in four possible forms within coal:

- *Surface moisture*: water held on the surface of coal particles or macerals.
- *Hydroscopic moisture*: water held by capillary action within the microfractures of the coal.
- *Decomposition moisture*: water held within the coal's decomposed organic compounds.
- *Mineral moisture*: water which comprises part of the crystal structure of hydrous silicates such as clays.

The results of proximate analysis are generally recorded as a percentage of the air-dried material.

4.2.1.1.1 Procedure

About 1 gram of finely powdered coal sample having size less than -212 micron was taken.The sample was placed on a watch glass.Watch glass was kept in an electric hot air oven. The oven was maintained at a temperature of $108^0 \pm 2^0$c.The sample was kept in the oven at 108^0c for 1.5 hours.After 1.5 hours sample was taken out and kept in the desiccator for about 10 minutes.Then the sample was weighed.Moisture content was calculated as per the following formula:

——*100

Where X = Weight of empty watch glass in gram

Y = Weight of watch glass + coal sample before heating in gram

Z = Weight of watch glass + coal sample after heating in gram.

4.2.1.2 Ash

Coal ash is the residue remaining after the combustion of coal under specified conditions. It does not occur as such in the coal but is formed as the result of chemical changes that take place in the mineral matter during the combustion process. These changes can be the loss of carbon dioxide

from carbonate, loss of water from silicate, conversion of iron pyrite to iron oxide. Ash and mineral matter of coal are therefore not identical.

There are two types of mineral matter present in coal. One is extraneous mineral matter while the other is inherent mineral matter. The extraneous mineral matter occurs due to

I. the association of minerals with the decaying vegetable matters while converting into coal. This type of mineral matter is difficult to remove because they are closely associated with the coal. So this type of mineral matter can't be removed by mechanical means.
II. Dirt, rock mixed up during the loading, transportation and handling of coal. This can be removed by mechanical means.

Inherent mineral matter is due to the inorganic elements associated with the plant materials from which the coal is formed.

4.2.1.2.1 Procedure

The crucible was cleaned by heating it in the muffle furnace for 1 hour at 800^0c. Then it was taken out and cooled to room temperature.The crucible was weighed.Approximately 1 gram of coal sample was taken.Sample was placed in the crucible which was then kept in the muffle furnace. The muffle furnace was maintained at 450^0c for 30 minutes and then at 850^0c for 1 hour. The crucible was taken out and cooled.Then the weight of the crucible was calculated.% Ash is calculated by the following formula:

$$\frac{\qquad}{\qquad}$$

Where X = weight of empty crucible in gram

Y = weight of coal sample + crucible before heating in gram

Z = weight of coal sample + crucible after heating in gram.

4.2.1.3 Volatile matter

The volatile matter of a coal is of particular importance in assessing the use for which the coal is suitable. Volatile matter in coal refers to the components of coal, except for moisture, which are liberated at high temperature in the absence of air. This is usually a mixture of short and long chain hydrocarbons, aromatic hydrocarbons, hydrogen, ammonia, tar vapour, oxygen containing

compounds, carbon monoxide and some sulfur. It is alsoessential to exclude air from the crucible during heating to preventoxidation and, therefore, the lid of the crucible is fit. Themoisture content of the sample should be determined at the same time asthe volatile matter so that volatile matter content can be calculated by subtracting it with the moisture content.

4.2.1.3.1 Procedure

A special volatile matted crucible having 38mm height, 25mm external diameter and 22mm internal diameter was used for the determination of volatile matter.The empty crucible was weighed.Approximately 1 gram of coal sample was placedin the crucible.The crucible was kept in the muffle furnace with its lid closed maintained at 925^0c.The crucible was kept in the furnace for exactly 7 minutes.Then the crucible was removed and allowed to cool.% of Volatile matter was calculated from the following formula:

Where X = weight of empty crucible in gram

Y = weight of coal sample + crucible before heating in gram

Z = weight of coal sample + crucible after heating in gram.

Figure 4.2: Photographic view of muffle furnace for Volatile matter and Ash content determination

4.2.1.4 Fixed carbon

The fixed carbon content of the coal is the carbon found in the material which is left after volatile materials are driven off. This differs from the ultimate carbon content of the coal because some carbon is lost in hydrocarbons with the volatiles. Fixed carbon is used as an estimate of the amount of coke that will be yielded from a sample of coal. Fixed carbon is determined by calculating the sum of the ash, moisture and volatile mattercalculated on air dried basis and then subtracting it from 100.

Fixed carbon = 100 - (Moisture + Volatile matter + Ash)

Table 4.2 Results of Proximate analysis of fresh coals

Sl No.	Sample	M(%)	VM(%)	A(%)	FC(%)
1	MCL-1	6.7	40.9	11.5	40.9
2	MCL-2	9.6	31.7	17	41.7
3	SECL-1	5	22.55	28.9	43.55
4	SECL-2	3.65	23.4	19.9	53.05
5	SECL-3	7.45	18.05	17.5	57
6	SECL-4	4.55	23.95	16.95	54.55
7	SECL-5	2.2	31.8	19.6	46.4
8	SECL-6	6	19.4	21.25	53.35
9	SCCL-1	7.5	22	24.85	45.65
10	SCCL-2	6.1	18.9	8.2	66.8
11	SCCL-3	7.15	28.55	18.15	46.15
12	CCL-1	2.85	20	37.45	39.7
13	CCL-2	3.6	20	6.5	69.9
14	INDO-1	6	27.6	9.1	57.3
15	INDO-2	5.2	24.7	11.2	58.9

Table 4.3 Results of VM content of 1 and 3 day oxidized coals

Sl No.	Sample	VM(%)	
		1 day	3 day
1	MCL-1	45.5	53.85
2	MCL-2	37.3	48.7
3	SECL-1	28.4	43.9
4	SECL-2	27.4	42.3
5	SECL-3	23.15	50.25
6	SECL-4	29.05	43.05
7	SECL-5	35.7	47.2
8	SECL-6	25.15	37.7
9	SCCL-1	26	35
10	SCCL-2	26.3	41.15
11	SCCL-3	34.25	41.65
12	CCL-1	24.65	31.25
13	CCL-2	25.2	34.7
14	INDO-1	32.65	43.2
15	INDO-2	29.85	39.9

4.2.1.5 GROSS CALORIFIC VALUE

It is the amount of heat released when unitmass of the coal is burnt in oxygen saturatedcondition. The residual products consist ofcarbon dioxide, sulphur dioxide, nitrogen and water. Gross calorific value of coal depends upon its chemical composition, which is governed by geological environment during its coalification process, mainly on its maturity.Combustible elements present in coal includecarbon,hydrogen, sulphur etc**.** Non-combustible elements include silica, iron, aluminum, calcium, magnesium etc. The gross calorific value is determined by bomb calorimeter.

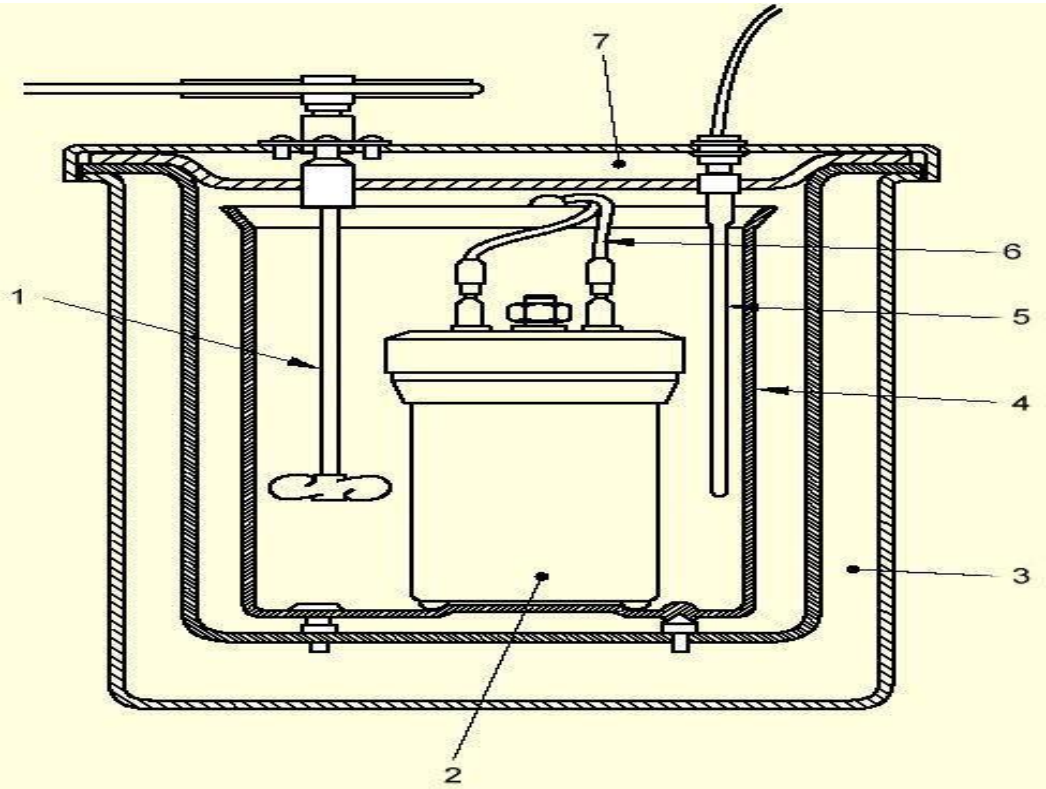


Figure 4.3 Schematic diagram of Bomb Calorimeter

1. Stirrer
2. Calorimeter bomb
3. Jacket
4. Calorimeter vessel
5. Platinum resistance thermometer
6. Ignition lead
7. Jacket

4.2.1.5.1 Bomb calorimeter

Bomb calorimeter consists of

- Bomb:

  It is a stout cylindrical chamber made of stainless steel. It is of 500 ml capacity and should be resistant to intergranular corrosion. It should be capable of withstanding an internal pressure of 25 atm without any permanent deformation. The cover of the bomb has three terminals, one for the entry of oxygen while the other two is for igniting the sample. Once the pressure is set in the bomb the terminal for entering of oxygen into the bomb is closed.

- Calorimeter:

  It should hold two litres of water with the bomb covered in water. The water level should be atleast 2cm above the lid of the bomb.

- Stirring arrangement:

  For stirring water in the calorimeter cell efficient stirring arrangement shall be there. The rate of stirring shall be kept at constant rate.

- Thermocouple:

  A thermocouple with sufficient arrangement to display temperature is given to display temperature digitally.

- Timer:

  A timer is there to display time so that reading can be taken at an appropriate interval.

- Crucible:

  A silica crucible of about 25mm diameter and 20mm height is used for keeping the coal pellet at place.

- Electrical circuit:

  Electrical circuit with supply of 6 volts is present to provide the ignition for the sample. The fuse wire is passed through the pellet.

- Water jacket:

  It is used to minimize heat loss by radiation. A stirrer is used for circulating the heat.

Figure 4.4 Photographic view of the bomb calorimeter apparatus

1. Calorimeter vessel
2. Tempereture reading unit
3. Stirrer
4. Firing cable

4.2.1.5.2 Procedure

Approximately 1 gram coal sample of -212 micron size was taken. A pellet was made from the coal and the weight of the pellet was measured.About 10cc of distilled water was taken in the bomb.The pellet was kept in the crucible and necessary arrangement was done by means of thread to keep contact between fuse wire and coal.A pressure of 25 atm was arranged in the bomb.2 litre of water was kept in the calorimeter vessel.Instrument was switched on.Stirring was done for 5 minutes.The initial temperature reading was taken.Then the bomb was fired. At first the temperature increased and then it became constant after reaching a maximum value. The maximum temperature reading was noted.

Water equivalent for the bomb used for experiment = 2411.5 Cal/^{0}c

Δt = Change in temperature

M = Mass of the pellet.

Table 4.4 Results of Calorific value of coal samples

Sl No.	Sample	Calorific Value (Kcal/kg)
1	MCL-1	4693.68
2	MCL-2	3733.78
3	SECL-1	4586.57
4	SECL-2	5198.17
5	SECL-3	4976.11
6	SECL-4	5644.47
7	SECL-5	5839.6
8	SECL-6	5220.5

9	SCCL-1	4827.66
10	SCCL-2	3693.48
11	SCCL-3	6265.17
12	CCL-1	5361.26
13	CCL-2	3862.16
14	INDO-1	4693.88
15	INDO-2	4417.4

4.3 SPONTANEOUS HEATING LIABILITY

4.3.1 CROSSING POINT TEMPERATURE

In this method the coal sample is heated in an oxidizing atmosphere. The lowest temperature at which the exothermic reaction in the coal bed is observed to be self-propellant under the experimental conditions is termed as CPT. In simple terms it is the temperature at which the bath temperature of bath and coal becomes equal.

4.3.1.1 Procedure

A coal bed of 20 gram of -212 micron sieve size was taken and kept in a reaction tube.The reaction tube was put in the glycerin bath.A constant air flow of 80 ml/minute was maintained.Two thermometers, one in the coal bed while the other in the glycerin bath, were placed.Then the assembly was heated at 1^0c per minute.A stirrer was provided in the glycerin bath for dissipation of heat in glycerin.One thermocouple was kept in the glycerin bath to show temperature while the other chromel-alumel thermocouple was kept in the reactor tube within coal.The temperature of both coal and glycerin was recorded at 5 minute interval till the temperature of the coal and glycerin bath became equal.

Table 4.5 Classification of coal's liability to spontaneous heating as per CPT

CPT(^{0}c)	Liability
120-140	High
140-160	Moderate
160-180	Low

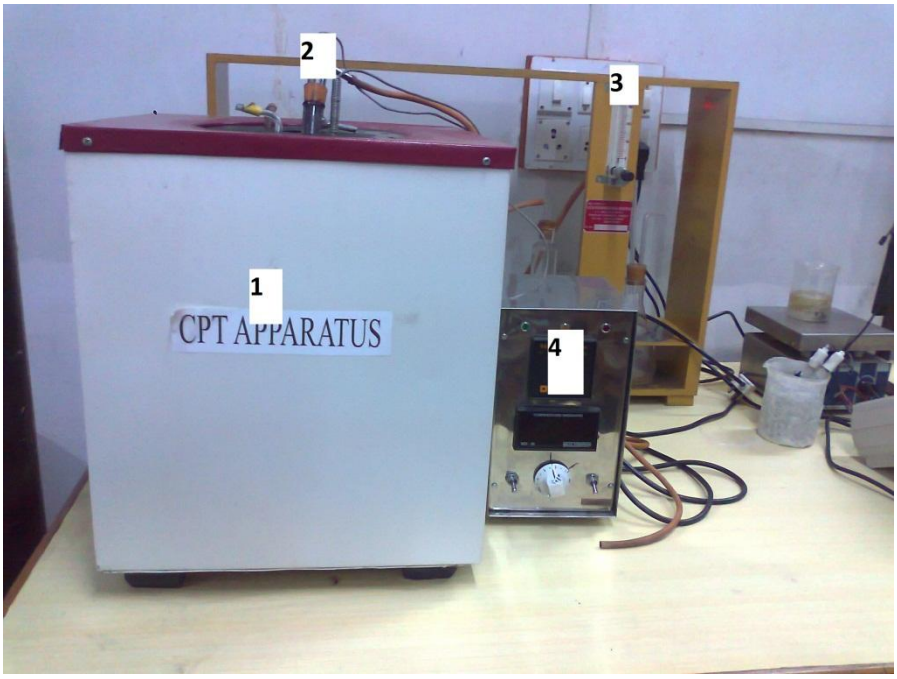


Figure 4.5 Photographic view of the CPT apparatus

1. Glycerene bath
2. Reaction tube
3. Flow meter
4. Tempereture indicator

Table 4.7 Results of CPT of fresh and 1 day oxidized coal samples

Sl No.	Sample	CPT(^{0}C)	CPT after 1 day oxidation(^{0}C)
1	MCL-1	164	234
2	MCL-2	165	236
3	SECL-1	160	235
4	SECL-2	167	243
5	SECL-3	157	227
6	SECL-4	163	232
7	SECL-5	161	235
8	SECL-6	172	235
9	SCCL-1	168	240
10	SCCL-2	165	240

11	SCCL-3	149	228
12	CCL-1	146	229
13	CCL-2	170	248
14	INDO-1	190	268
15	INDO-2	146	227

4.3.2 Wet oxidation potential

The coal molecules consist of two parts: the aliphatic or hydro-aromatic structure and condensed aromatic structure. From these aliphatic hydrocarbons are more prone to oxidation which condensed hydrocarbons are resistant to oxidation. Hydroxyl group gives a very high degree of reactivity to coal as hydroxyl group in the aromatic structure gets oxidized faster. Low rank coals are rich in such hydroxyl groups therefore low rank coals are rather easily oxidized. Due to smaller extent of condensed aromatic structures in them lower rank coals on oxidation produce large amounts of aliphatic acids compared to higher rank coals. Since the high rank coals have structure close to that of graphite, it is less liable to oxidation and the products contain more aromatics than aliphatics. It is generally seen that lower the potential difference, lower is the susceptibility of coal to spontaneous heating.

In this experiment potential measurements were made between a saturated calomel electrode and a carbon electrode immersed in the coal oxidant mixture in a suspension of 0.5 g coal powder in a solution containing 1 normal potassium hydroxide and deci normal potassium permanganate solution at the ambient temperature. The potential difference between carbon electrode and calomel electrode inserted in the solution is measured at 1 minute interval for 40 minutes. A magnetic stirrer is given for continuous stirring of the solution. The potential difference between the initial and final solution gives a measure of susceptibility to spontaneous heating. Low difference indicates high susceptibility while high difference shows low susceptibility.

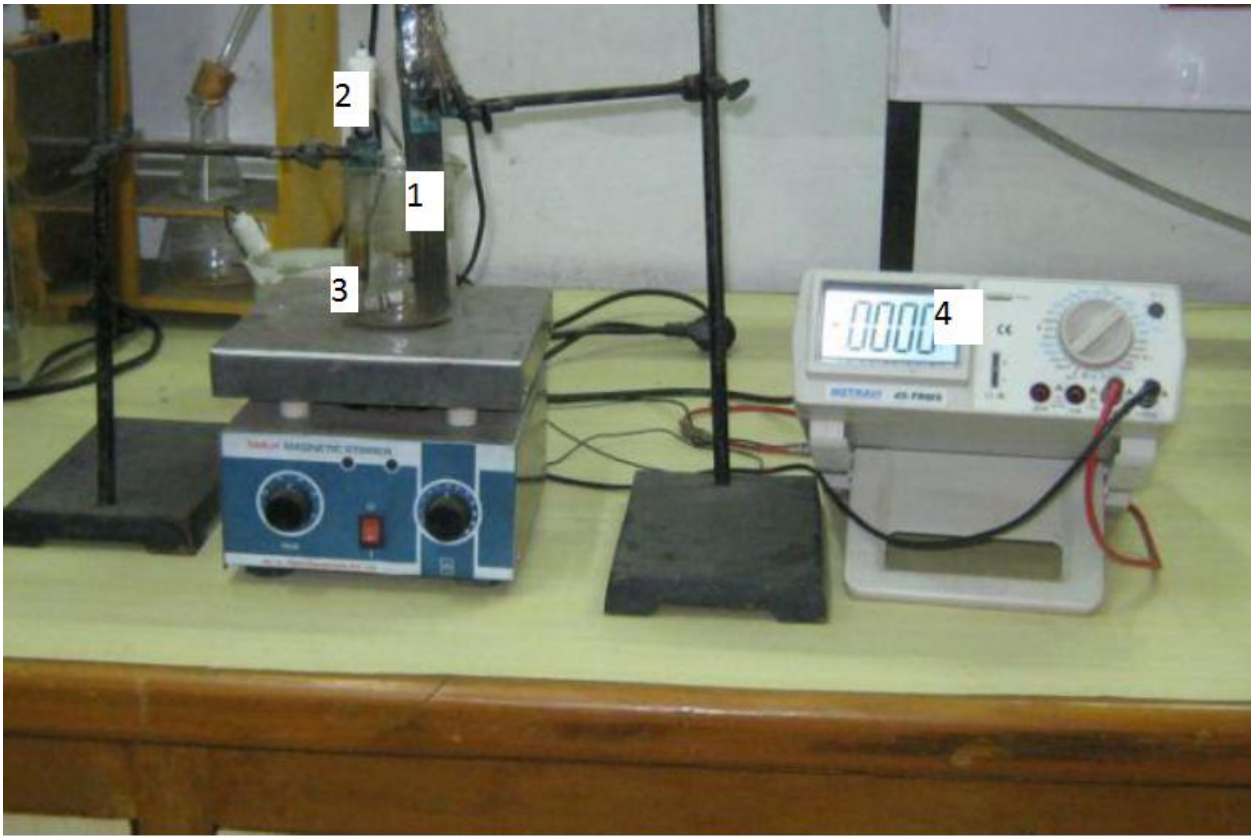


Figure 4.6 Photographic view of the Wet Oxidation Potential setup used for experiment

1. Carbon electrode
2. Calomel electrode
3. Magnetic stirrer
4. Voltage reading unit

4.3.2.1 Procedure

First 100ml of solution of 1N KOH and 0.1N $KMnO_4$ was taken. A magnetic stirrer was put in the solution for complete mixing of solution. 0.5 gram of coal sample was added to the solution. A carbon electrode along with a calomel electrode was put into the solution. The potential difference was measured between the two electrodes in a millivoltmeter. The potential difference was noted at 1 minute interval for 40 minutes. The difference between the initial and final potential difference was calculated.

Table 4.8 Results of wet oxidation potential of fresh, 1 day oxidized and 3 day oxidized coal samples

Sl No.	Sample	EMF(mV) of fresh coal	EMF(mv) after 1 day	EMF(mv) after 3 day
1	MCL-1	82.4	67	54.7
2	MCL-2	88.7	75.9	58.3
3	SECL-1	93.4	78.2	53.3
4	SECL-2	89.2	79.6	63.5
5	SECL-3	93.2	73.6	50.8
6	SECL-4	90	64.2	60
7	SECL-5	83.7	76.5	65.5
8	SECL-6	84.9	78.5	63.2
9	SCCL-1	81.6	66.2	56.8
10	SCCL-2	82.4	69.2	46.6
11	SCCL-3	86.8	73.9	49.6
12	CCL-1	113.5	89.4	64.9
13	CCL-2	82.6	64.5	56.3
14	INDO-1	75	66.1	51.3
15	INDO-2	118	93.6	64.2

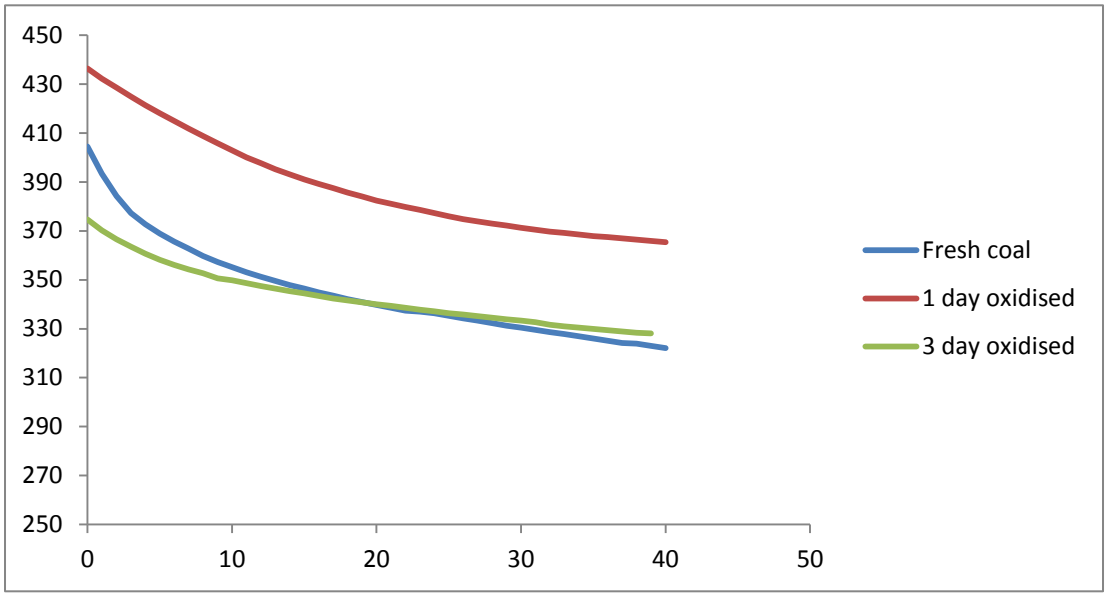


Figure 4.7 Wet oxidation potential curve of SECL 5 sample

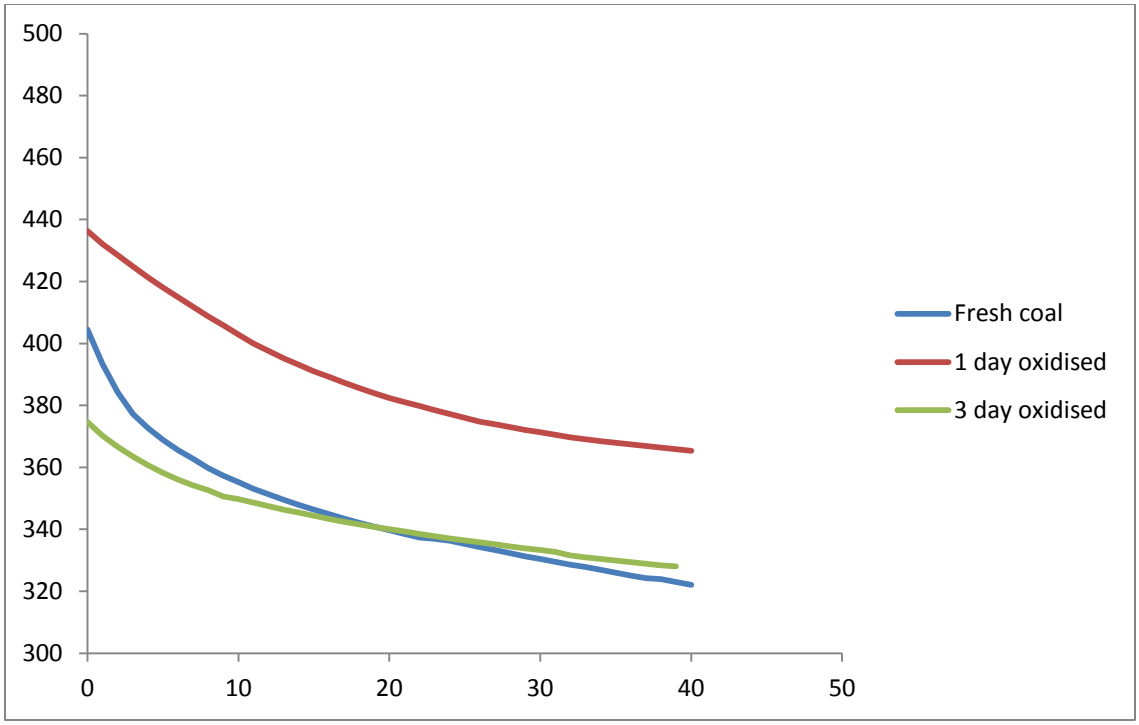


Figure 4.8 Wet oxidation potential curve of SECL 3 sample

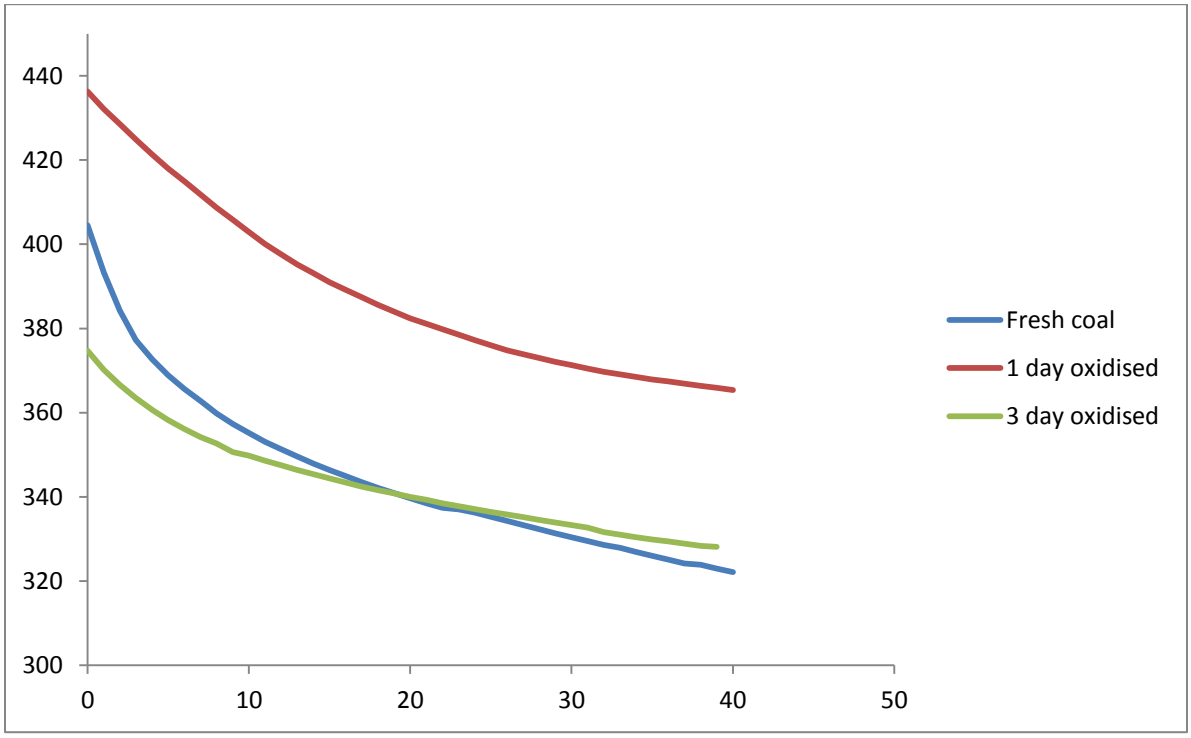


Figure 4.9 Wet oxidation potential curve of SECL 1 sample

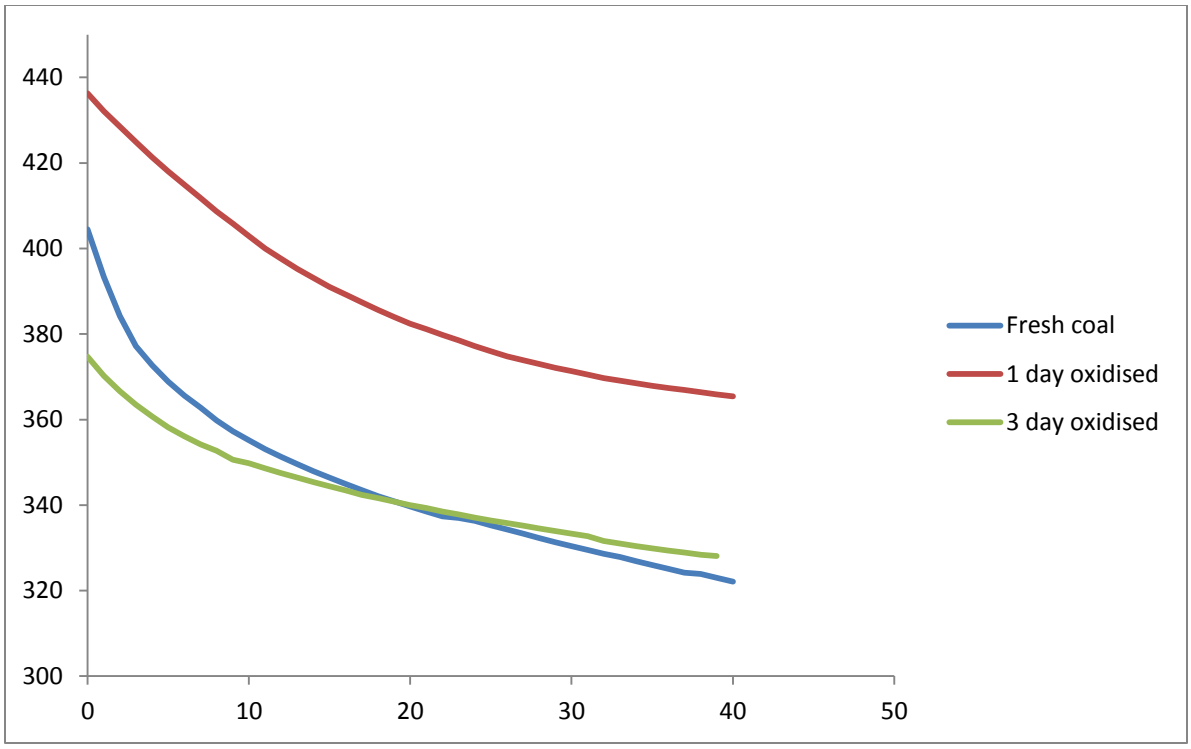


Figure 4.10 Wet oxidation potential curve of INDO 2 sample

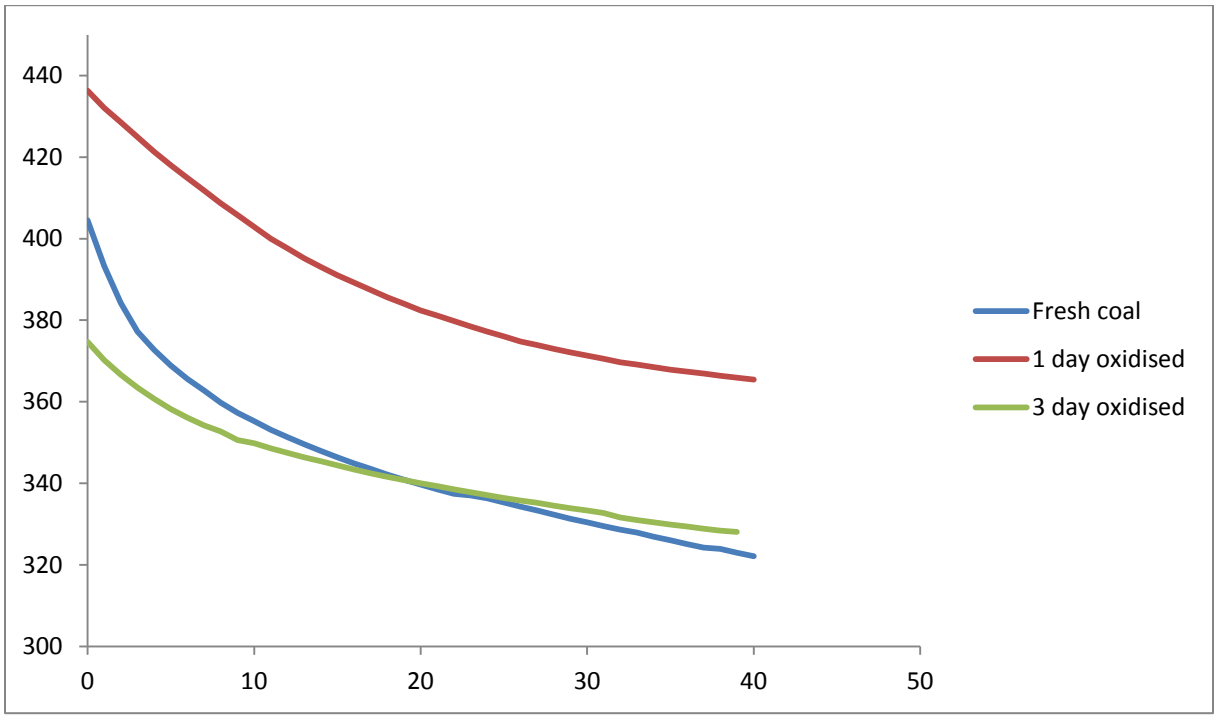


Figure 4.11 Wet oxidation potential curve of CCL 2 sample

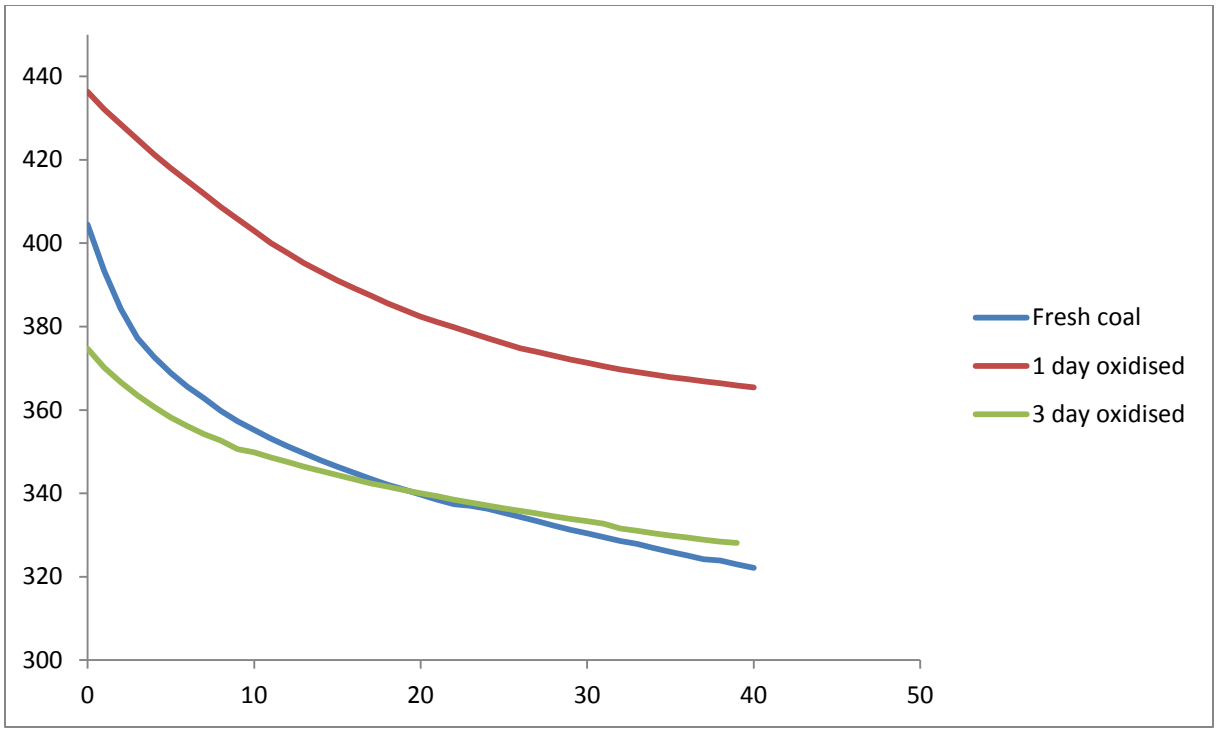


Figure 4.12 Wet oxidation potential curve of SECL 2 sample

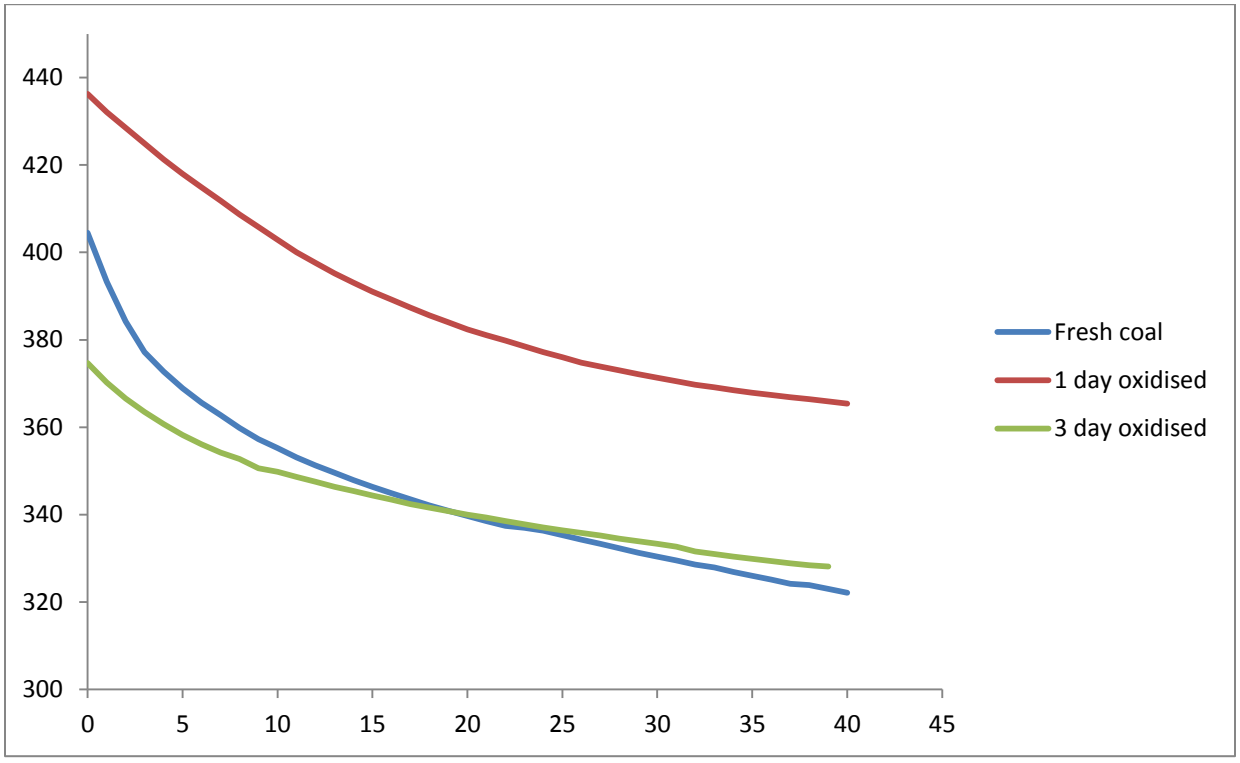


Figure 4.13 Wet oxidation potential curve of SCCL 2 sample

4.3.3 Differential thermal analysis

Thermal analysis is the technique by which physical and chemical changes of a substance are measured as a function of temperature as the substance is subjected to a controlled heating rate. In this method the temperature difference between a coal sample and a thermally inert reference material is recorded against temperature as the two specimens are subjected to identical temperature changes in a block which is heated at a linear heating rate. If a sample undergoes no thermal change and the temperature difference is zero, then a base line parallel to recording instrument is given by the recording instrument. If the sample undergoes a thermal decomposition reaction with loss or gain of heat then the temperature gradient with respect to inert material will change producing a positive or negative ΔT depending on whether the transformation taking place in the sample is endothermic or exothermic. The curve recorded with ΔT plotted on the ordinate and temperature or time on the abscissa is called the DTA curve or thermogram.

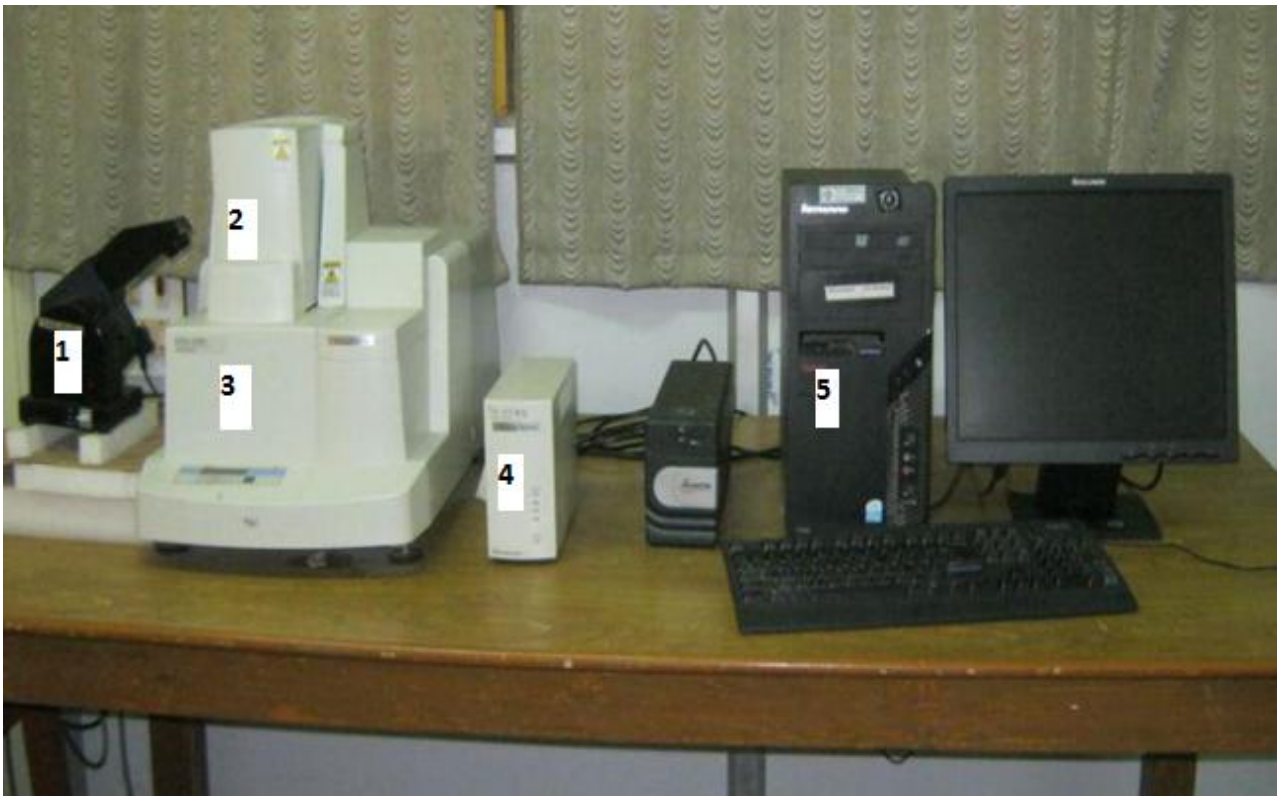


Figure 4.14 Photographic view of DTA setup used for experiment

1. Blower
2. Tubular furnace
3. Differential thermal analyzer
4. TA-60WS collection monitor
5. Computer

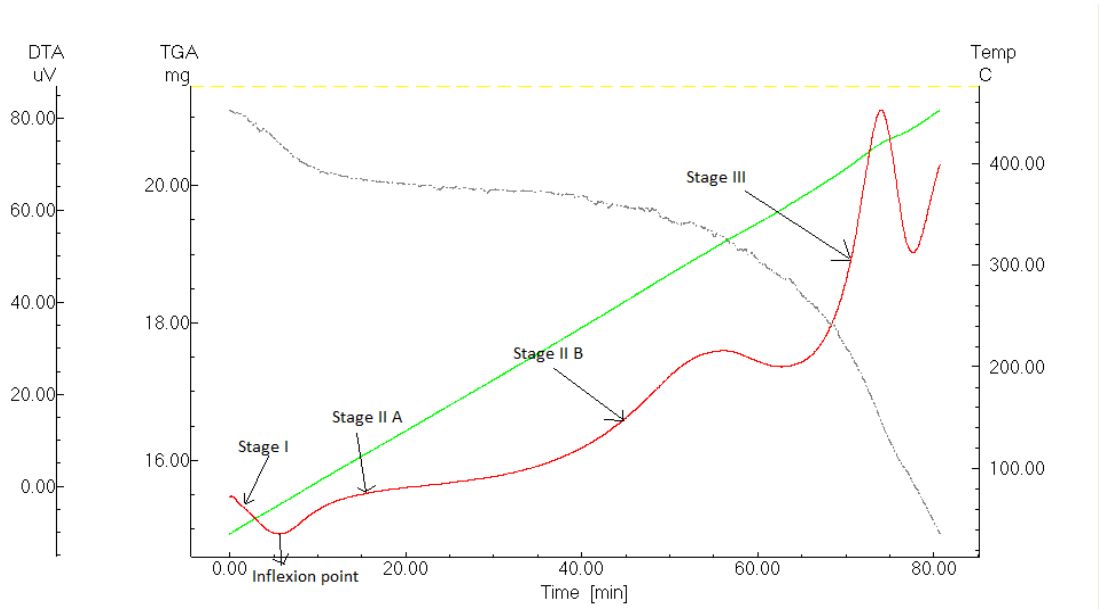


Figure 4.15 DTA thermogram for INDO 1 sample

The plot consists of three parts: stage I, II, III. During stage I, mostly endothermic reactions take place and the temperature falls due to release of moisture. Stage II consists of two parts, IIA and IIB. From the beginning of IIA the heating tendency is accompanied by a small amount of endothermic reactions. The point where IIA begins is called as the inflexion point. In IIB exothermic reactions start to dominate. The beginning of stage III is called as Transition temperature. This temperature is very important as it is directly related to spontaneous heating tendency of coal. The lower the transition temperature, higher is the susceptibility of coal to spontaneous combustion and vice versa. From stage III onwards complete exothermic reactions occur and the temperature continues to rise steeply. The slope of the DTA thermogram is also taken as indicators of spontaneous combustion. Lower the slope values lower is the susceptibility. The transition temperature was found out by drawing tangents at the inflexion point and the highest slope stage III. Their intersection gave the transition temperature.

Table 4.8 Transition temperatures obtained from DTA thermogram

Sample name		Transition temperature
INDO 1	Fresh	187.04
	1 day oxidized	227.39
	3 day oxidized	267.24
SECL 1	Fresh	162.91
	1 day oxidized	392.97
	3 day oxidized	401.81
SECL 2	Fresh	185.07
	1 day oxidized	383.99
	3 day oxidized	406.3

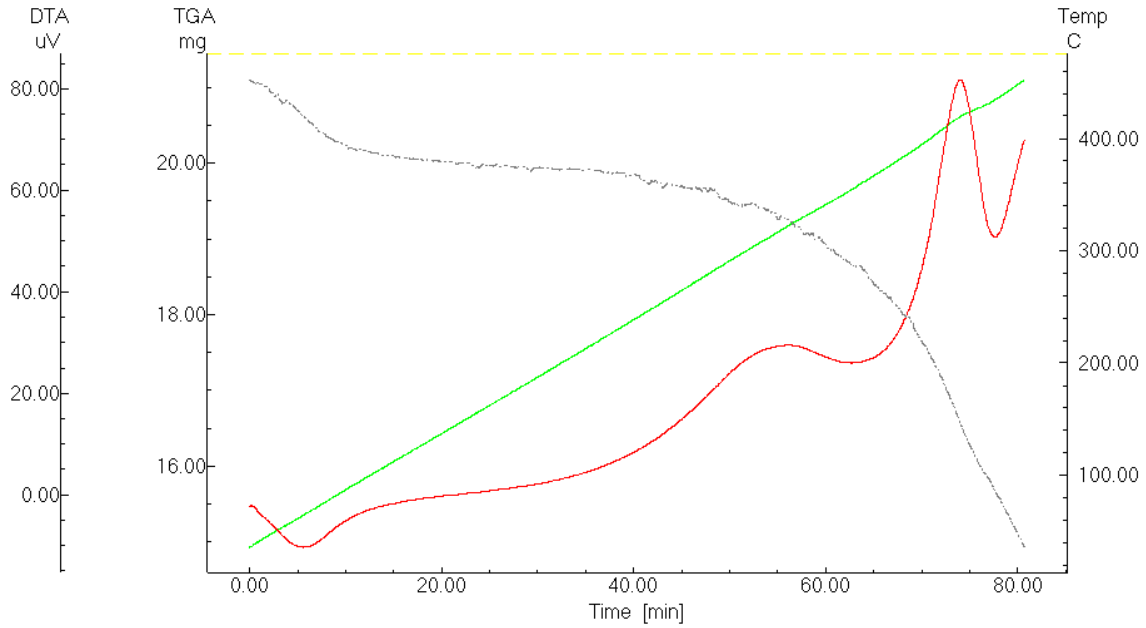


Figure 4.16 DTA Thermogram of INDO 1 fresh coal sample

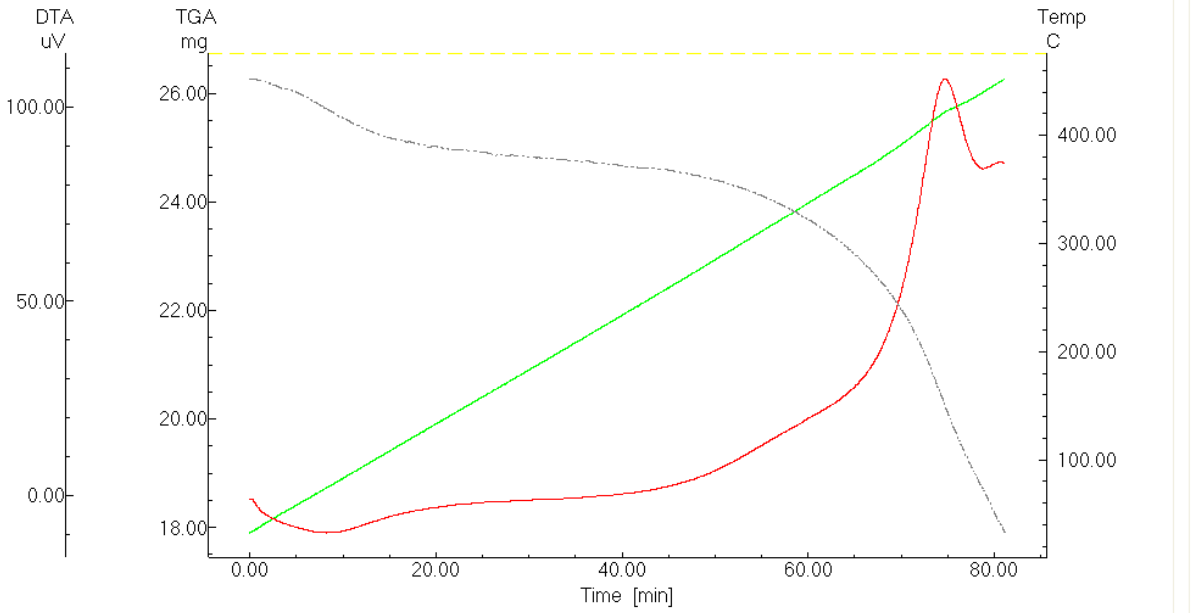


Figure 4.17 DTA Thermogram of INDO 1 1 day oxidized coal sample

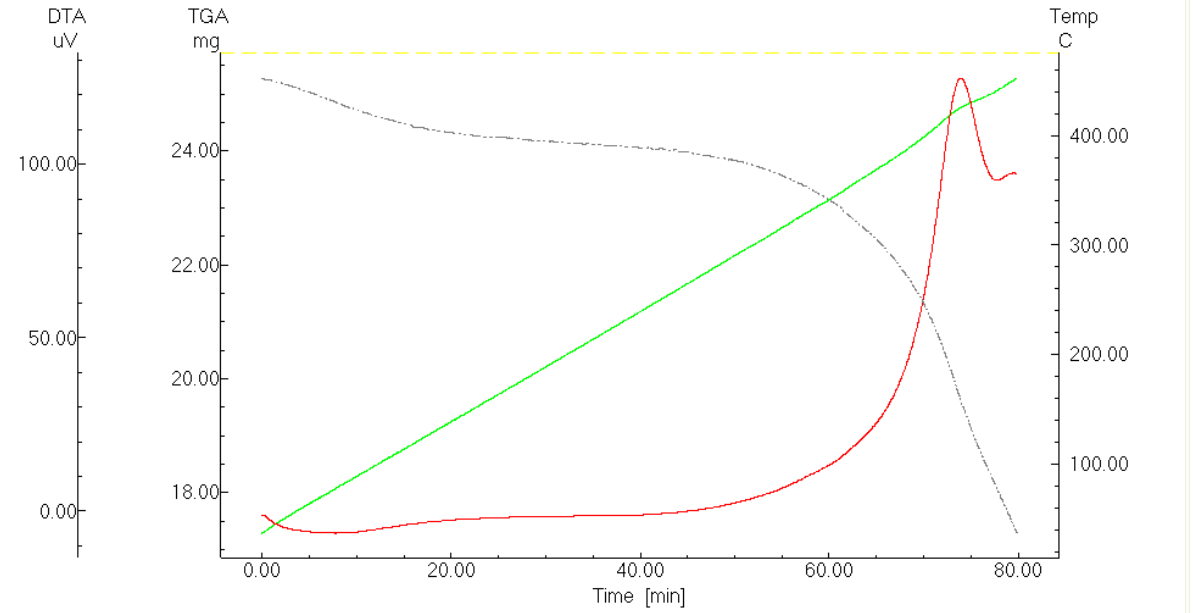


Figure 4.18 DTA Thermogram of INDO 1 3 day oxidized coal sample

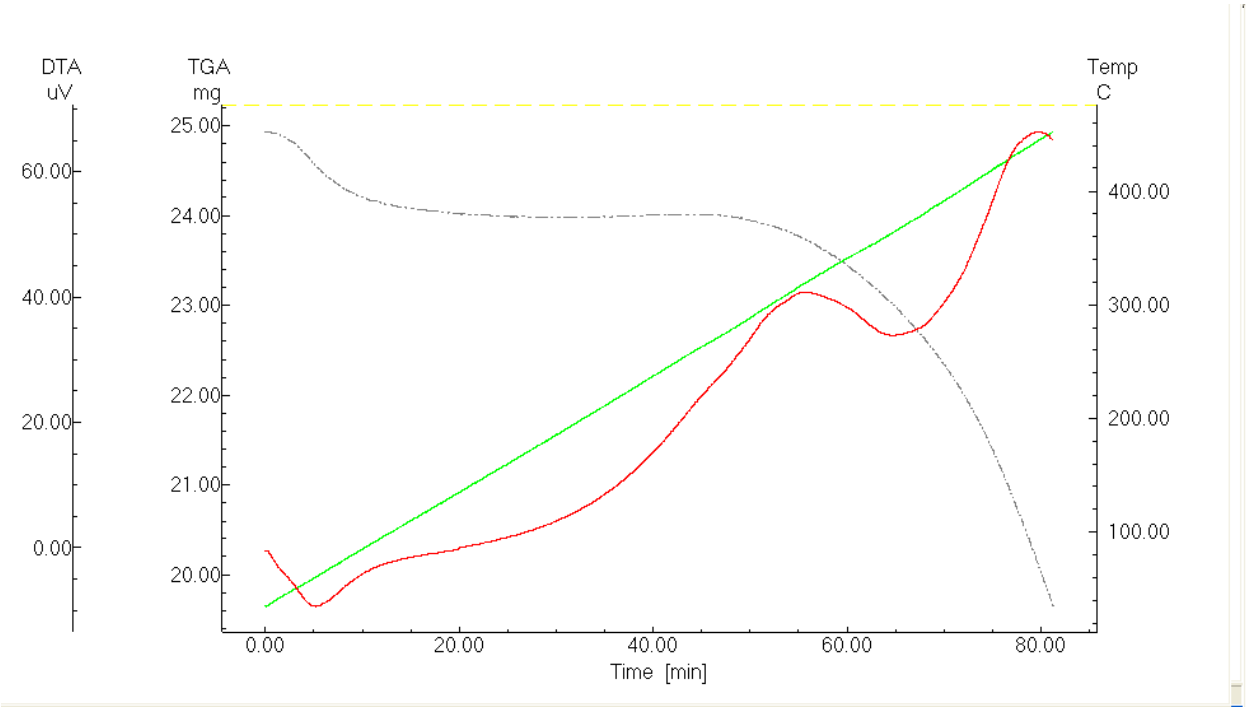


Figure 4.19 DTA Thermogram of SECL 1 fresh coal sample

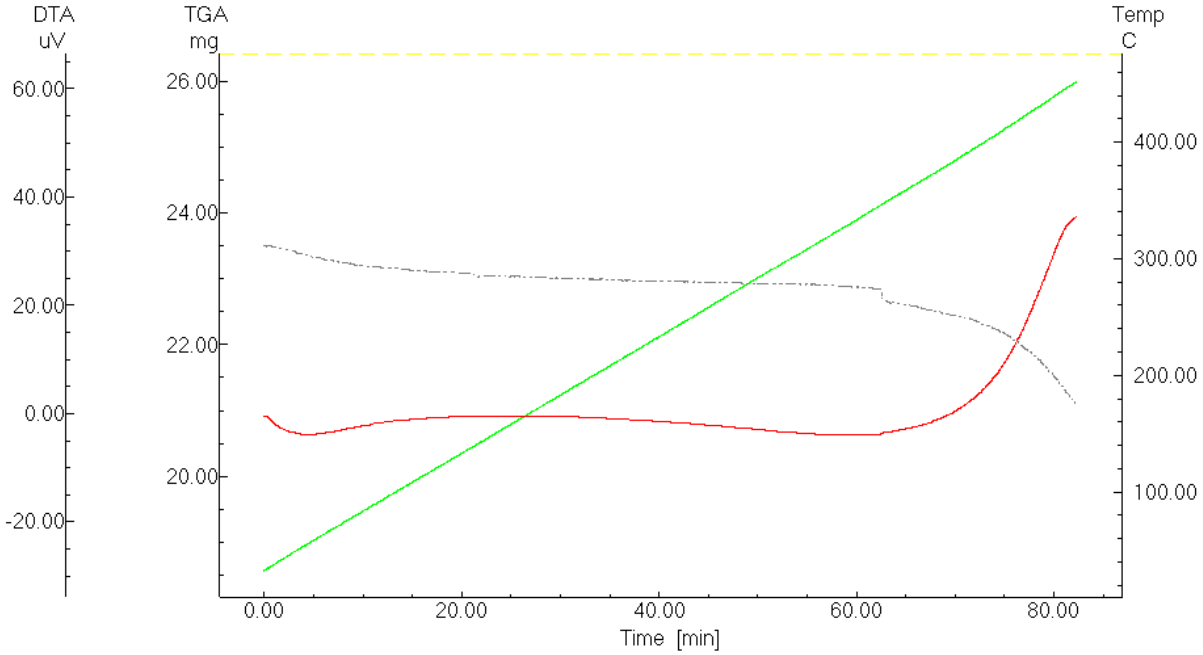


Figure 4.20 DTA Thermogram of SECL 1 1 day oxidezed coal sample

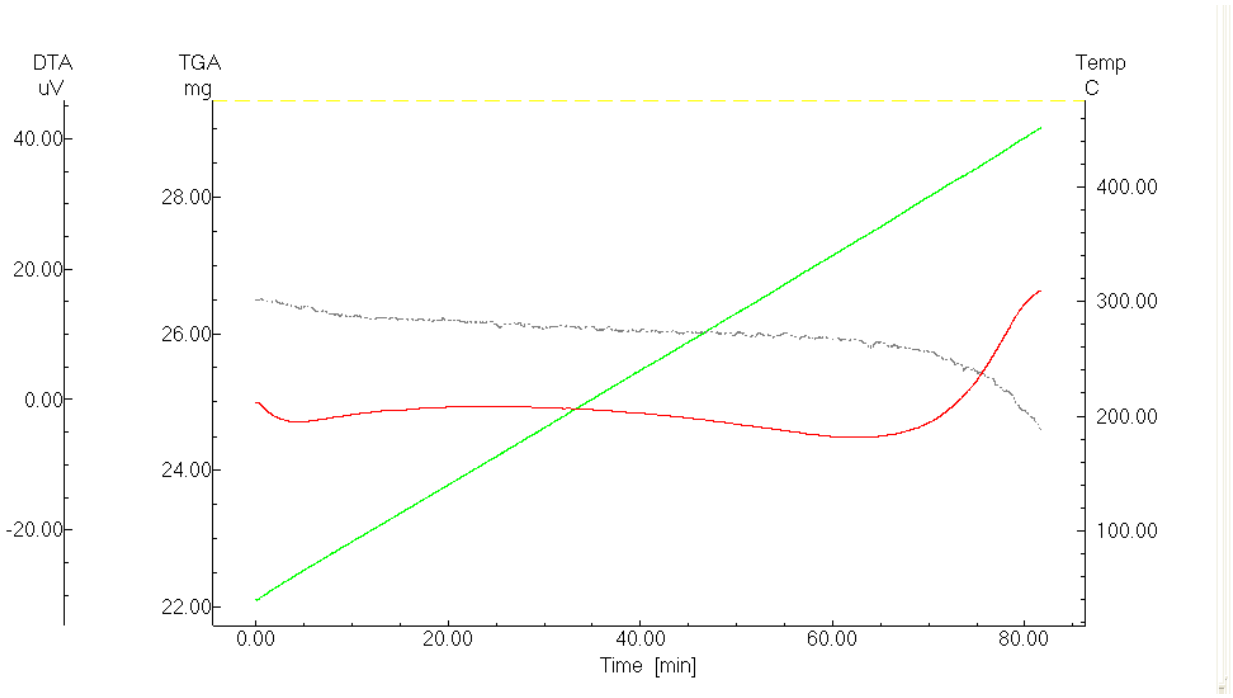


Figure 4.21 DTA Thermogram of SECL 1 3 day oxidized coal sample

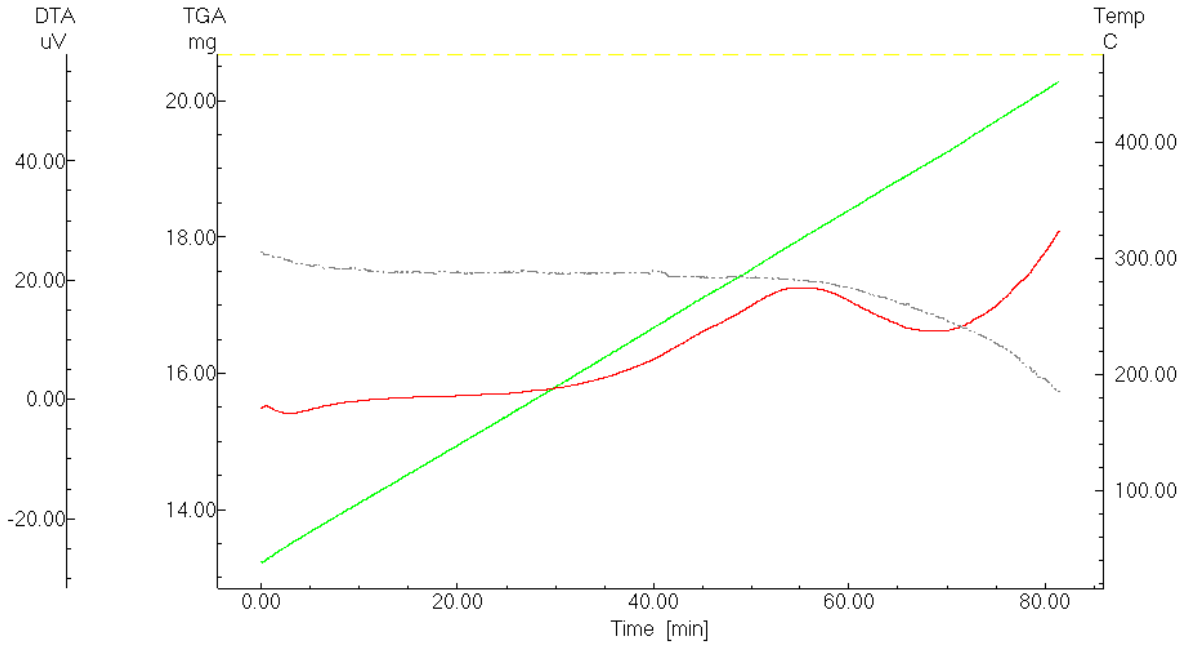


Figure 4.22 DTA Thermogram of SECL 2 fresh coal sample

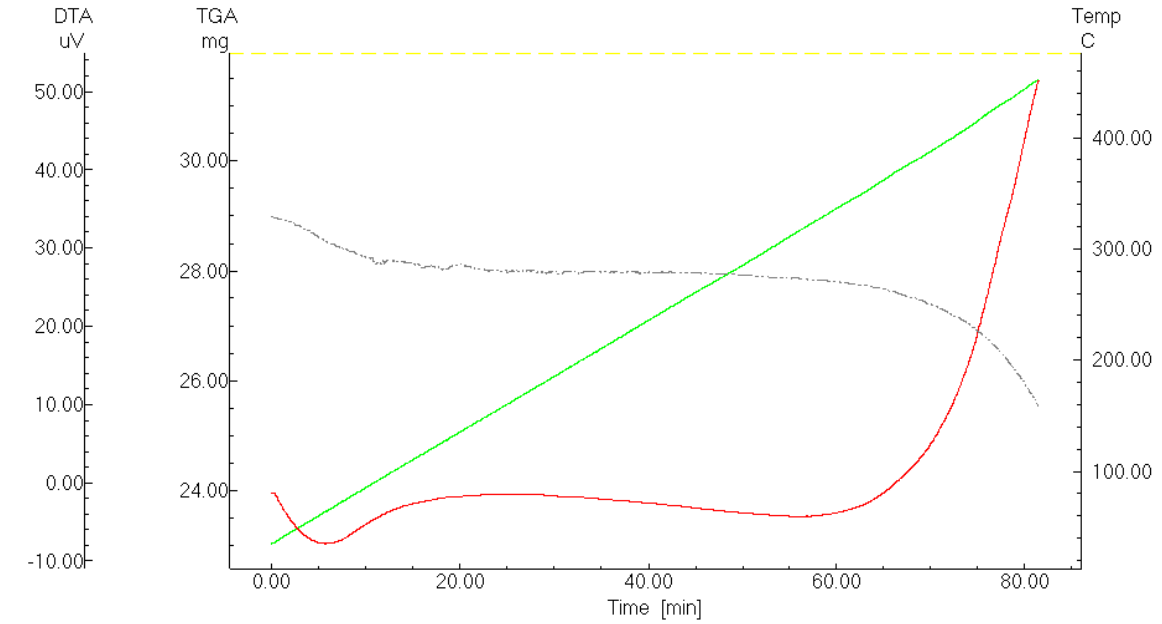


Figure 4.23 DTA Thermogram of SECL 2 1 day oxidized coal sample

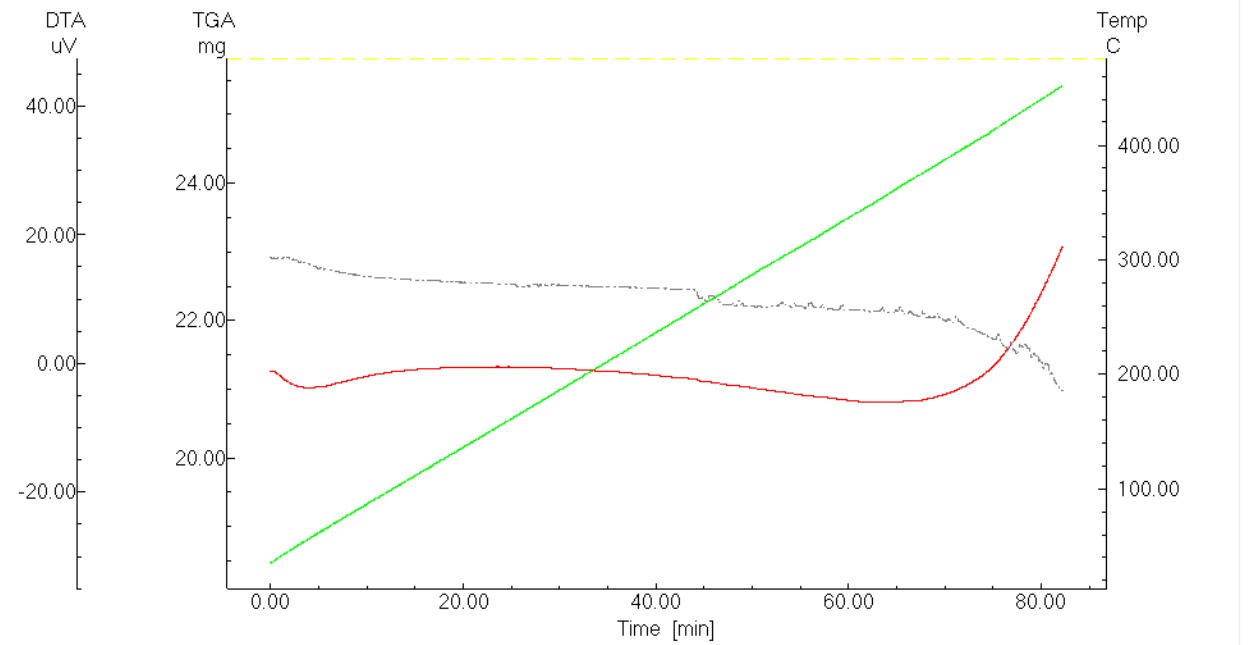


Figure 4.24 DTA Thermogram of SECL 2 3 day oxidized coal sample

CHAPTER 5

CORRELATION STUDY

CHAPTER 5

5. CORRELATION STUDY

Correlation studies were carried out to find out the relationship between intrinsic property of the coal sample and the spontaneous heating liability. These correlation studies were carried out individually for each property and different spontaneous heating liability such as CPT, Wet Oxidation potential. In addition to that the change in liability of coal to spontaneous heating with the change in oxidation period is analyzed through correlation. Along with these studies empirical relations are also established between these parameters by plotting the independent variable in the X direction while the dependent variable in Y direction. The intrinsic properties taken into consideration are moisture, volatile matter, ash and calorific value.

Table 5.1 Correlation between CPT and Intrinsic properties

Sl no.	Independent Variable	Empirical relation	R value
1	Moisture (M)	$CPT=0.0623M^5-1.1625M^4+15.763M^3-71.249M^2+152.91M+33.135$	0.3238
2	Ash(A)	$CPT=-0.0069A^3+0.4372A^2-8.5745A+213.84$	0.5578
3	Volatile Matter (VM)	$CPT = 0.0006VM^5 - 0.0828VM^4 + 4.4927VM^3-119.58VM^2 + 1562.5VM - 7864.1$	0.1915
4	Calorific Value (CV)	$CPT = -8E\text{-}15CV^5 + 2E\text{-}10CV^4 - 2E\text{-}06CV^3+0.0108CV^2 - 27.092CV + 27038$	0.4034

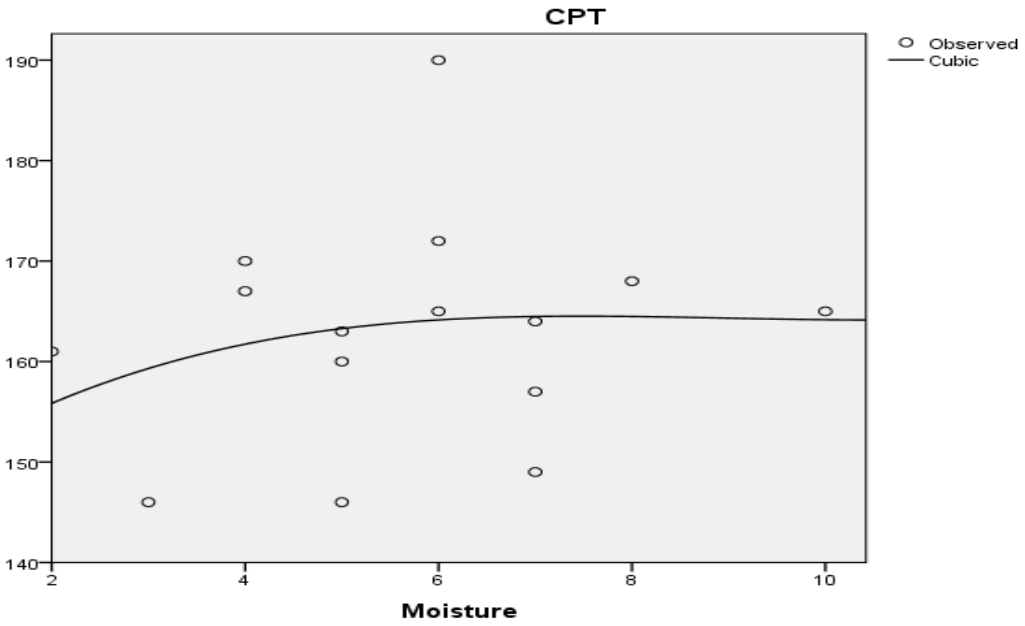


Figure 5.1 Correlation plot between CPT and Moisture content

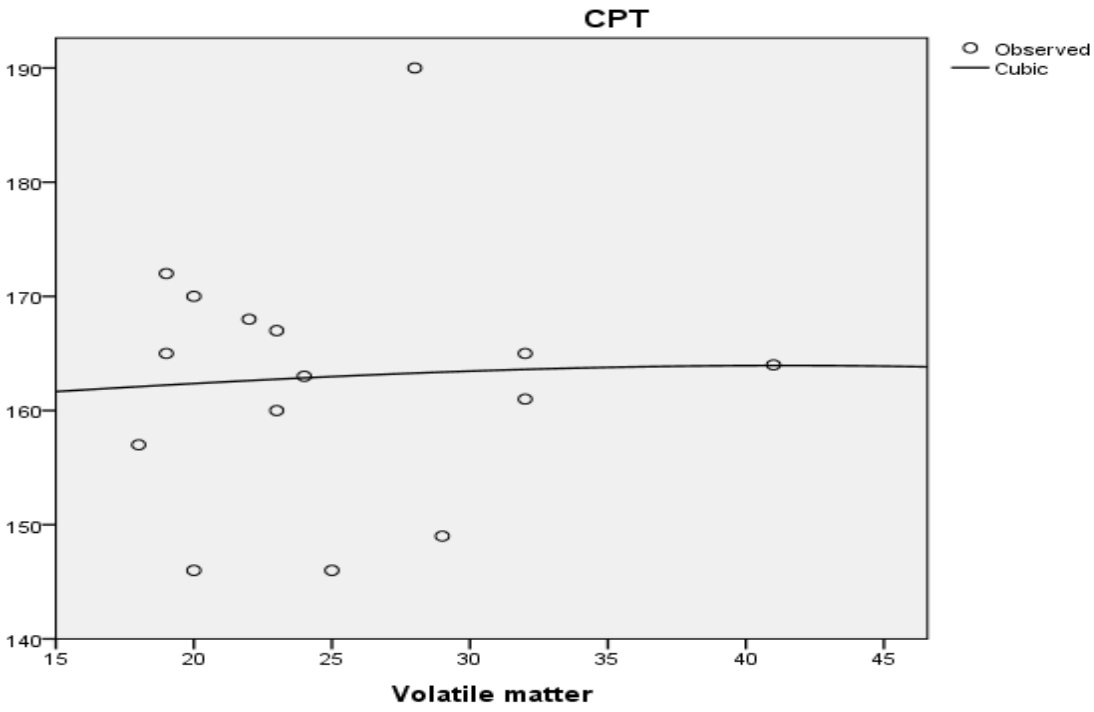


Figure 5.2 Correlation plot between CPT and Volatile matter

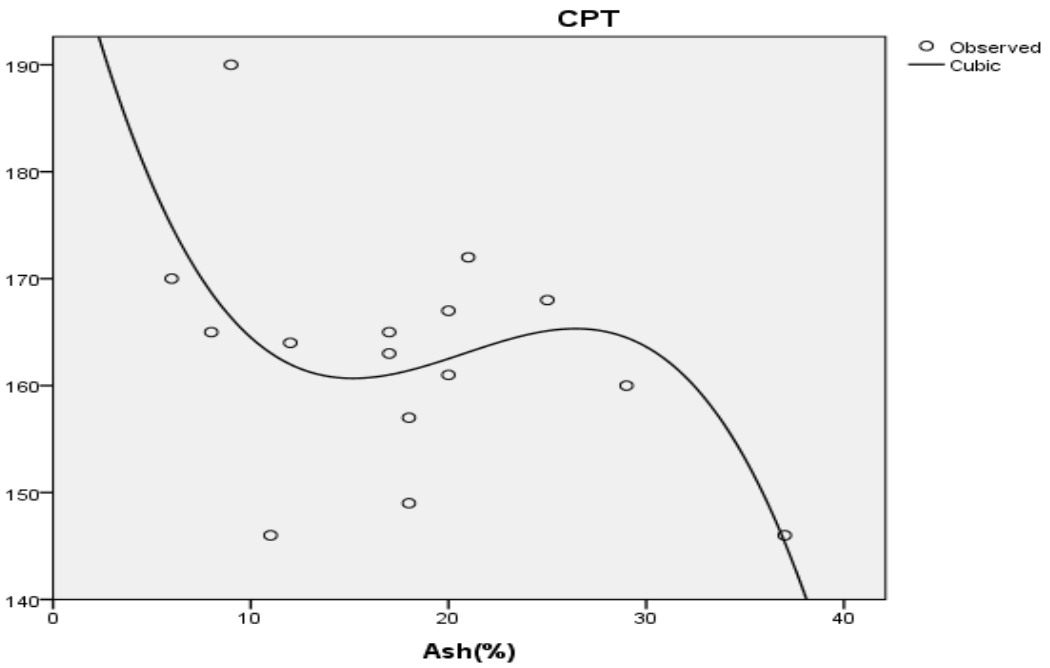


Figure 5.3 Correlation plot between CPT and Ash Content

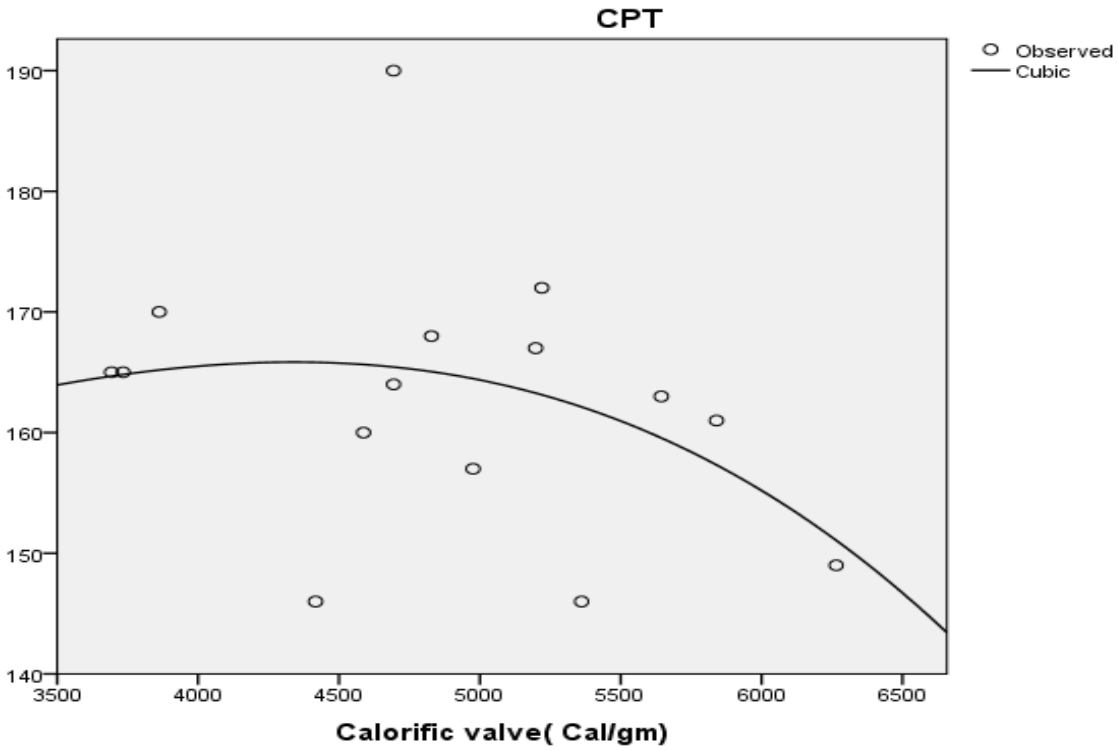


Figure 5.4 Correlation plot between CPT and Calorific Value

Table 5.2 Correlation between wet oxidation potential and intrinsic properties

Sl no.	Independent Variable	Empirical relation	R value
1	Moisture (M)	$WOP=0.292M^3+5.073M^2+25.242M+56.531$	0.3487
2	Ash(A)	$WOP=0.0077A^3-0.4607A^2-8.3484A+43.83$	0.6432
3	Volatile Matter (VM)	$WOP=-0.0008VM^5+0.1155VM^4- 6.2676VM^3 + 166.41VM^2 - 2163.9VM+ 11126$	0.3893
4	Calorific Value(CV)	$WOP = 2E\text{-}14CV^5 - 5E\text{-}10CV^4 + 5E\text{-}06CV^3 - 0.0254CV^2 + 63.341CV – 62644$	0.3757

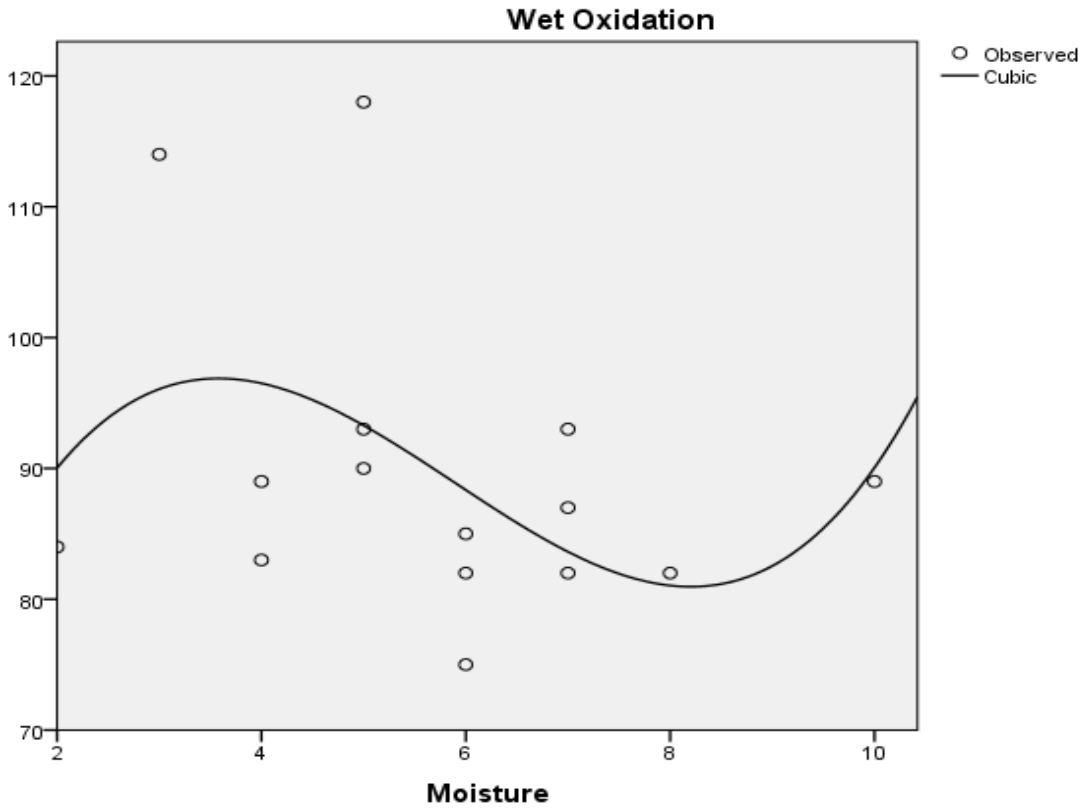


Figure 5.5 Correlation plot between Wet Oxidation Potential and Moisture content

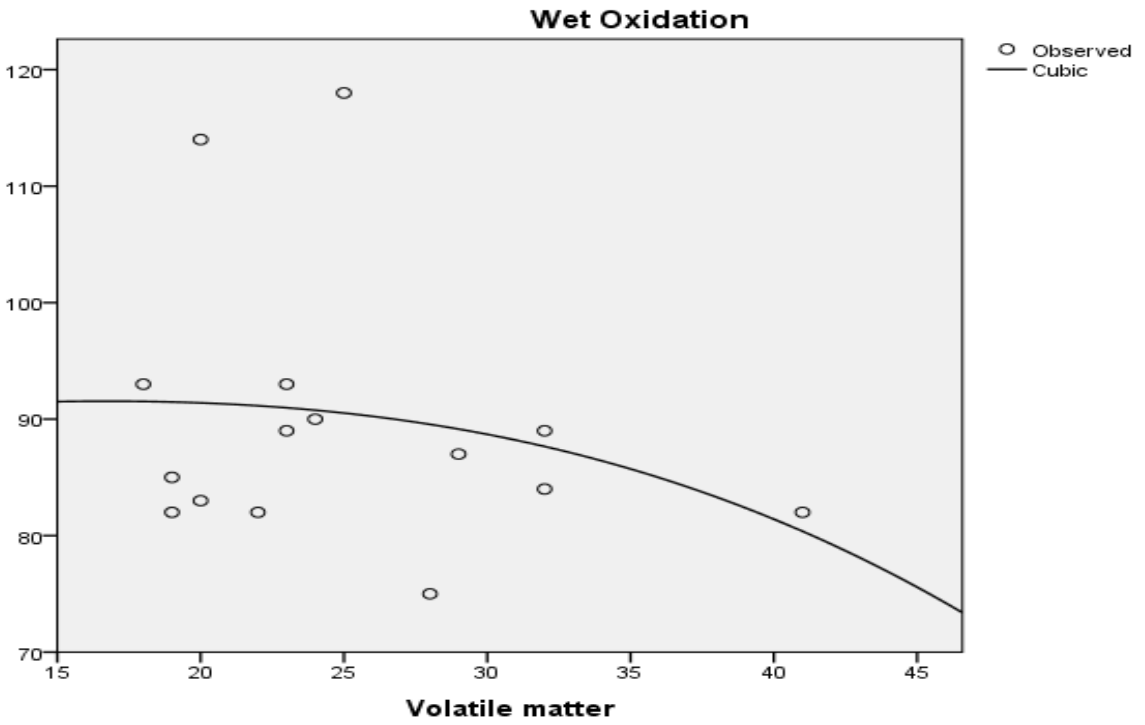


Figure 5.6 Correlation plot between Wet Oxidation Potential and Volatile matter

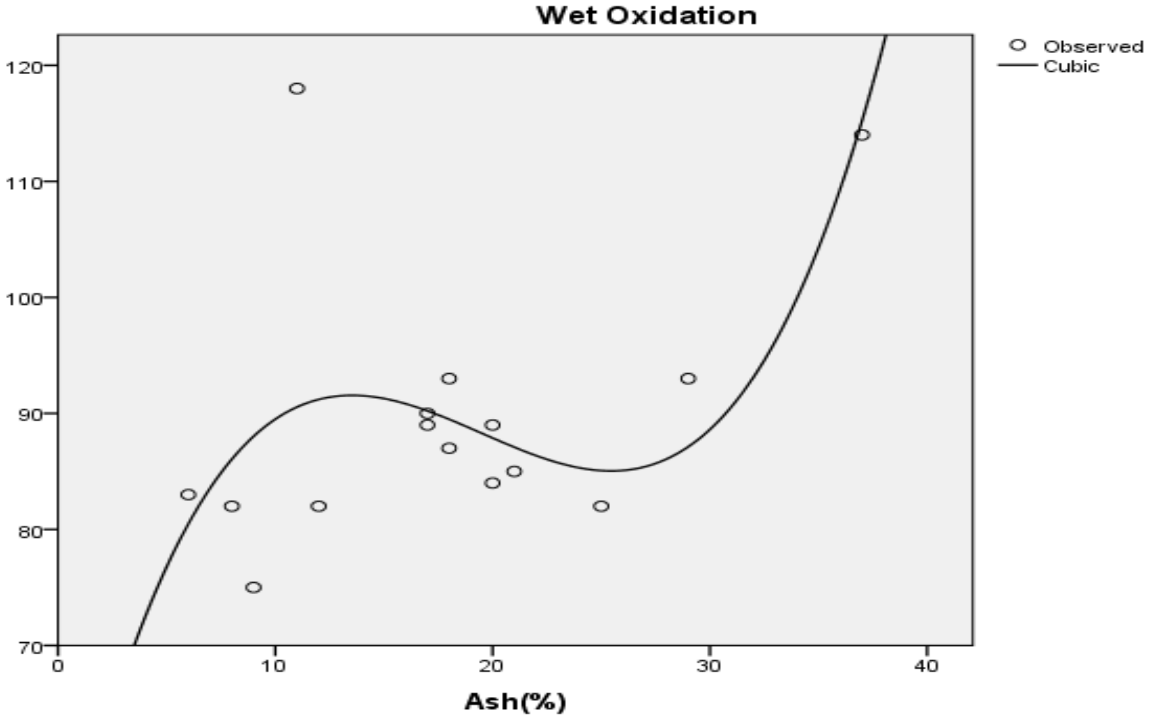


Figure 5.7 Correlation plot between Wet Oxidation Potential and Ash content

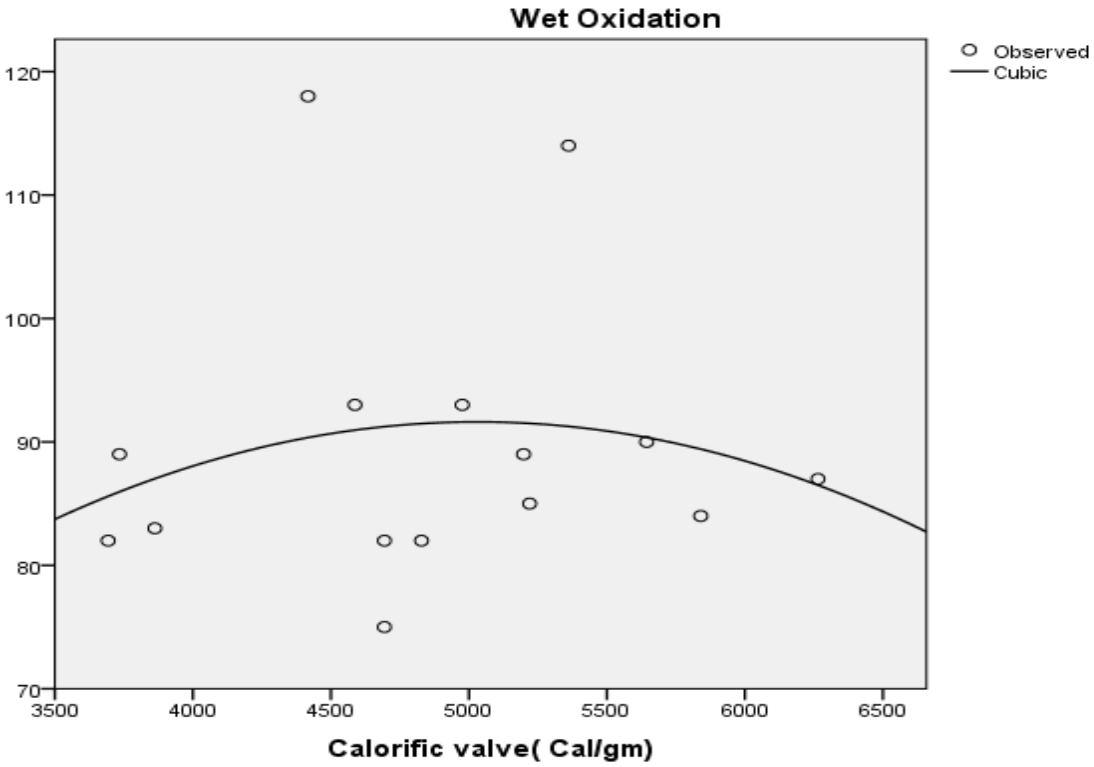


Figure 5.8 Correlation plot between Wet Oxidation Potential and Calorific Value

Table 5.3 Correlation between CPT and Wet Oxidation Potential of 1 day oxidized coal sample with volatile matter content

Sl no.	Independent Variable	Empirical relation	R value
1	Volatile Matter (VM)	$CPT^* = 0.0002VM^4 – 0.0168VM^3 + 0.5721VM^2 – 7.5638VM + 268.65$	0.141
2	Volatile Matter(VM)	$WOP^* = -0.002VM^4 + 0.2278VM^3 - 9.6731VM^2 + 178.24VM - 1126.2$	0.35

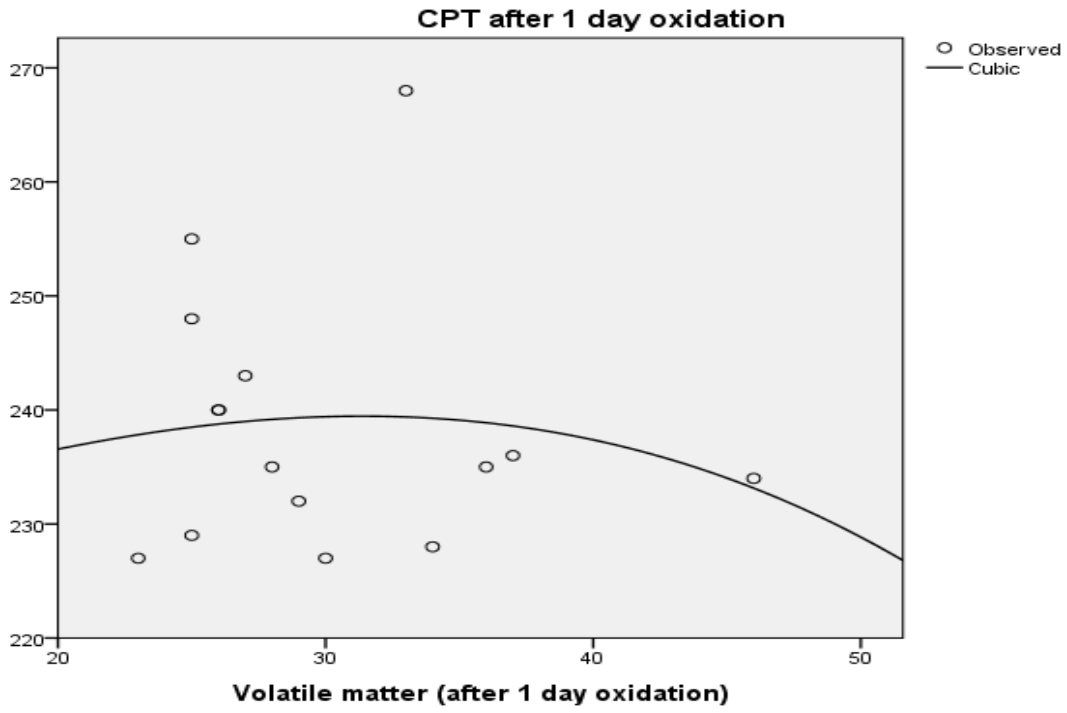


Figure 5.9 Correlation plot between CPT and Volatile matter content of a 1 day oxidized coal sample

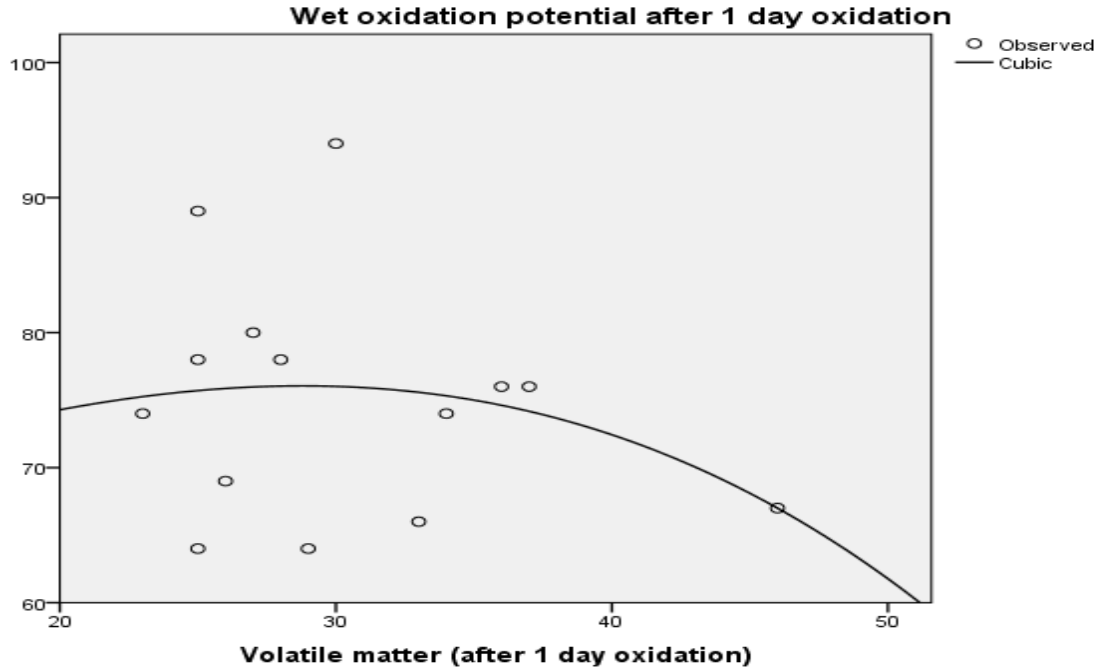


Figure 5.10 Correlation plot between Wet Oxidation Potential and Volatile matter content of a 1 day oxidized sample

Table 5.4 Correlation between Wet Oxidation Potential of 3 day Oxidized coal sample and Volatile matter

Sl no.	Independent Variable	Empirical relation	R value
1	Volatile Matter(VM)	$WOP^{***}=0.0007VM^5 - 0.142VM^4 + 11.829VM^3-487.18VM^2+9913.8VM - 79674$	0.6067

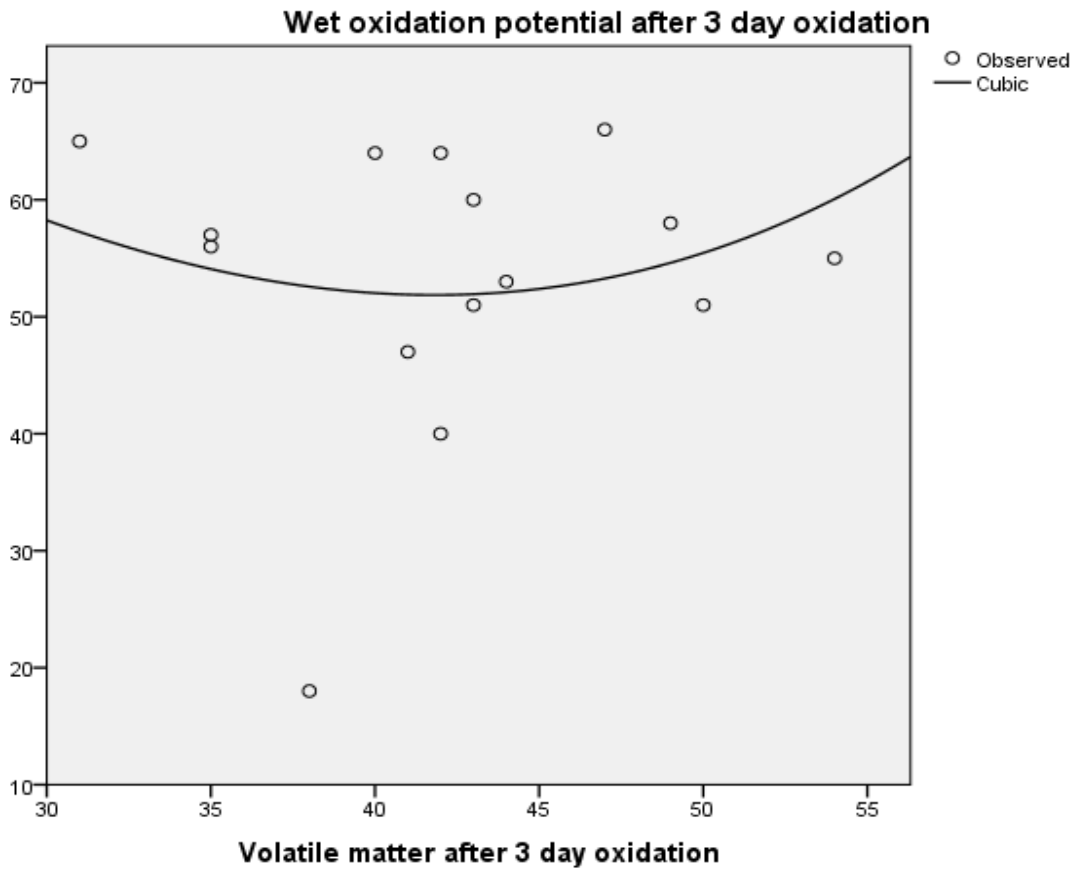


Figure 5.11 Correlation plot between Wet Oxidation Potential and Volatile matter content of a 3 day oxidized sample

CHAPTER 6

DISCUSSION AND CONCLUSION

CHAPTER 6

6.DISCUSSION AND CONCLUSION

6.1 DISCUSSION

To assess the spontaneous heating tendency of both fresh and oxidized coals, 15 coal samples were collected from different coalfields of the country. Among these coal samples 6 were from SECL, 2 from MCL, 2 from CCL, 3 from SCCL.2 samples were from Indonesia to compare its characteristics with Indian coal. One third portion of the sample was oxidized at 200^0c for 1 day and another one third was oxidized at 200^0c for 3 day. Oxidized coal sample was prepared from fresh coal sample by putting in a muffle furnace maintained at 200^0C. Intrinsic property of all of the fresh and oxidized coal samples were determined by proximate analysis and calorific value using a bomb calorimeter.The spontaneous heating tendency were determined by using crossing point5 temperature, wet oxidation potential and differential thermal analysis techniques. Finally correlation studies were carried out to establish relationship between these parameters and to see the change in intrinsic property and liability to spontaneous heating with increase in oxidation period.

It may be observed from table 4.2 that the moisture content of the coal sample varied from 2.2 to 9.6. SECL 5 coal sample has minimum 2.2 percentage moisture content while MCL 2 coal sample showed maximum moisture content of 9.6%. The volatile matter content varies from 18.05 (SECL 3) to 40.9 (MCL 1). The ash content varied from 9.1 to 37.45%.

Air oxidation of coal produces an increase in the volatile matter content. As the oxidation period increases the volatile matter content also increased. It may be observed from table 4.3 thatthe VM content in SECL-5 coal sample increased from 23.95% of fresh coal to 29.05% of 1 day oxidized coal sample. With the increase in oxidation period i.e. 3 days the volatile matter reached a value of 43.05. This may be due to removal of moisture.

Spontaneous heating liability was determined from CPT, DTA and wet oxidation potential.As the crossing point temperature decreases the liability of coal to spontaneous heating increases. In case of DTA with thedecrease in transition temperature the susceptibility of coal to spontaneous heating decreases.In wet oxidation potential experiment the potential difference occurring after a certain period of time gives a measure of the coal's susceptibility to Spontaneous heating. If the potential difference is more then it indicates more susceptibility.

It may be observed from table 4.7 that INDO-2 and CCL-1 show the highest susceptibility as they have the lowest crossing point temperature i.e. 146^0c. The observation is verified through wet oxidation potential experiment. These two samples show the highest potential difference of 118 and 113.5 mv.

As the coal sample is oxidized, more the liability of the sample to spontaneous heating decreases. This can be verified table 4.7, 4.8 and 4.9. For instance it can be seen from table 4.7 that the CPT value of the coal increased from 172^0c to 255^0c for SECL-6 sample. Similarly from table 4.8, it can be seen that the potential difference for SECL 1 sample from wet oxidation potential experiment decreases from 93.4mv to 78.2 mv with 1 day oxidation and to 53.3 mv with 3 day oxidation.

From the table 4.9, it may be seen that transition temperature is lowest (162.97) for SECL 1 sample. This shows that SECL 1 sample is more prone to spontaneous heating in comparison to INDO 1 and SECL 2 sample. This is also proved from both the CPT and Wet oxidation potential result. It may also be seen from the table 4.9 that as the sample is oxidized the transition temperature decreases showing more proneness to spontaneous heating.

The relationship between spontaneous heating liability of coals with its intrinsic properties was analyzed by correlation studies between them. The summarized values of correlation coefficients are given below

Table 6.1 Summarized values of correlation coefficient between susceptibility index and intrinsic properties of fresh coal

Sl no.	Intrinsic properties	CPT(^{0}c)	Wet oxidation potential
1	M (%)	0.3238	0.3487
2	A (%)	0.5578	0.6432
3	VM (%)	0.1915	0.3893
4	CV	0.4034	0.3757

6.2 CONCLUSION

From the previous discussion the following conclusion can be drawn.

- CCL 1 and INDO 2 are moderately prone to spontaneous heating. Other samples are less prone to spontaneous heating.
- Ash content and CPT showed a good correlation but other intrinsic properties didn't show good correlation. So CPT cannot always be taken as a reliable method for measurement of spontaneous heating.
- Wet oxidation potential value showed good correlation value with ash content. However the r value for other properties is not so high. Therefore wet oxidation potential cannot be considered as a suitable method for measurement of spontaneous heating.
- By comparing the intrinsic properties of Indian coal with Indonesian coal it can be concluded that Indonesian coal has high fixed carbon percentage and low ash content compared to Indian coal.
- The correlation was poor for CPT with VM content for oxidized coals. However, wet oxidation potential difference showed a better correlation with the intrinsic properties.
- For coals with high fixed carbon content the change in liability of spontaneous heating is significant even after 1 day oxidation. While in case of coals with low fixed carbon content the change in liability of spontaneous heating is not too significant.

The empirical relationships obtained from the best fitted trend line don't show very good correlation. This can be attributed to the fact that some extreme points are present in our sample. The best way to avoid this will be to increase the number of samples for the correlation plot. As the number of sample increases the correlation plot will also increase.

CHAPTER 7

REFERENCES

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7.REFERENCES

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